Correlations between microstructure, nitrogen concentration and micro hardness of nitrided Ferritic Stainless Steel

by

Prepared by: Muhammad Azwan Ibrahim (ID: 13208)

Supervised by: Assoc. Prof. Dr. Patthi bin Hussain

Dissertation submitted in partial fulfillment of

The requirement for the

Bachelor of Engineering (Hons)

(Mechanical)

JANUARY 2015

Universiti Teknologi PETRONAS

Bandar Seri Iskandar

31750 Tronoh

Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

Of Research Project

CORRELATIONS BETWEEN MICROSTRUCTURE, NITROGEN CONCENTRATION AND MICRO HARDNESS OF NITRIDED FERRITIC STAINLESS STEELS

by

Muhammad Azwan Ibrahim

13208

A project dissertation submitted to the Mechanical Engineering Programme,
Universiti Teknologi PETRONAS in partial fulfillment of the requirement for the
BACHELOR OF ENGINEERING (Hons) MECHANICAL ENGINEERING

Approve by,

…….

(ASSOC. PROF. DR. PATTHI BIN HUSSAIN)

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained here have not been undertaken or done by unspecified sources or person.

Muhammad Azwan Ibrahim

13208

ABSTRACT

Gas Nitriding of Ferritic Stainless steels AISI 430 was carried out at 600℃, for various period of nitriding process which were for 2 hours, 8 hours and 24 hours. The effect of nitriding process period in term of microstructure, overall micro hardness, cross section hardness and nitrogen diffusion rate were investigated. The microstructures of samples were carried out using both Optical Microscope (OM) and Scanning Electron Microscope (SEM). The phase changes and Nitride precipitation formation were observed with respect to period of nitriding. Aside Microstructure, Energy Dispersive Spectroscopy (EDS) was conducted on the samples to investigate the nitrogen concentration inside the material. Vickers Hardness test have been carried out to investigate the effect of phase changes and it correlation toward the hardness of the materials.Unnitrided ferritic stainess steel having bigger size of grain boundaries were compared to nitride ferritic stainless steels and this is due to heat treatment during manufacturing process and concentration of nitrogen inside the steel.

The size of grain boundaries reduced with nitriding time due to result of nitrogen inside the steels. Unnitrided Ferritic stainless steel has the lowest hardness around 165.67Hv follow by Nitrided Ferritic stainless steel at 2 hours, 8 hours and 24 hours, each having 182.6Hv, 224.69Hv and 226.34Hv, respectively. The hardness value and concentration of nitrogen inside the steel reduces with the depth under the surface. The concentration of nitrogen in the steel increases with time of nitriding, highest concentration of nitrogen detected in 24 hours nitride stainless steel which approximately around 3.02%. Increasing in nitriding time increases the diffusion of nitrogen into the steel, thus improves the microstructure of the material, significantly increase the hardness properties of the materials.

ACKNOWLEDGEMENT

First of all, my greatest gratitude to Allah s w t for his blessing opportunity, and chances given along the process, provide me health, and ability to continue studies, and moral support to completing this Final Year Project within two semester. During the process I have been generous support from several person who contribute directly and indirectly toward completing this report, especially to my beloved parents Ibrahim Bin Lateh, Fatimah @Fatimah Sulha bt Hitam, pray for me every all the time, provide me allowance along final year studies.

I would also take this opportunity to express my deep gratitude and regards to my supervisor, Assoc. Prof. Dr. Patthi Hussain for his knowledge sharing, supervision intensive care supportive thought completing this project. The dedication and help given throughout the journey will be appreciated deeply, only Allah can fringe benefits thought this journey.

I also would like to take this opportunity to express my gratitude to Mr Hassan Mahmud and Mrs NurulHuda for their guidance, supportive and advices in helping me finishing this project especially in conduct nitriding process and result discussion. Im also would like to thanks to the technicians especially to Mr Danial and Mr Harris for their help and guidance, assistances, willingly spending time with me in the period of completing this project. This project ould not be able to be completed without this support and guidance.

Lastly special thanks to all my colleagues and friends especially my senior Wan Kamarussadam, Nazirul Nazlan and my project partner Ahmad Ikram for their support, knowledge and encouragements thought the process.

TABLE OF CONTENT

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1
INTRODUCTION

1.1 Background

1.1.1 Modern Steel Production

Steel originally in form of rust/iron ore such as **Hematite** (Fe_2O_3) and **Magnetite** (Fe_3O_4). The iron ore, being an iron oxide compound, cannot transform to metallic iron until the oxygen is removed or reduced from the compound. [1] Iron oxide is stable mode of iron particle after react with oxygen. The iron ore is then fed into a blast furnace, which act as chemical reactor to purify the iron ore with help from other substance such as coke and limestone producing pure iron. Impure steel goes in the top and come out at the bottom called **Pig Iron**. [1]

Coke and lime stone enhance the process oxidation and purifying the iron oxide. The coke in the furnace serve as a source of heat at the same time act as the source of reducing gas that will chemically separate oxygen atoms from iron oxide. Meanwhile, **limestone** used to act as purifying agent support in removing impurities other then oxygen from iron oxide. [1] Oxygen also needs to be supplied to support the combustion of the coke, regularly the oxygen came from surrounding air.

Below is the chemical reaction during the oxidation process [1]:

1. C (Coke) + $O_2 \longrightarrow CO_2$
2. $CO_2 + C \longrightarrow 2CO$
3. $2Fe_2O_3 + 3C \longrightarrow 4Fe + 3CO_2$
4. $Fe_2O_3 + 3CO \longrightarrow 2Fe + 3CO_2$

However, the iron produced from furnace is roughly are not pure due to high carbon content which is not suitable for making engineering material, because of this high content of carbon pig iron extremely brittle and weak. This Pig Iron must be further refined in order to be used. There are few type of furnace have been used to produced Pig Iron and also to produce steel from Pig Iron (Steel refining). **Blast Furnace** and **Direct reduction process** (DRI) mainly be used to produce Pig Iron. [1]

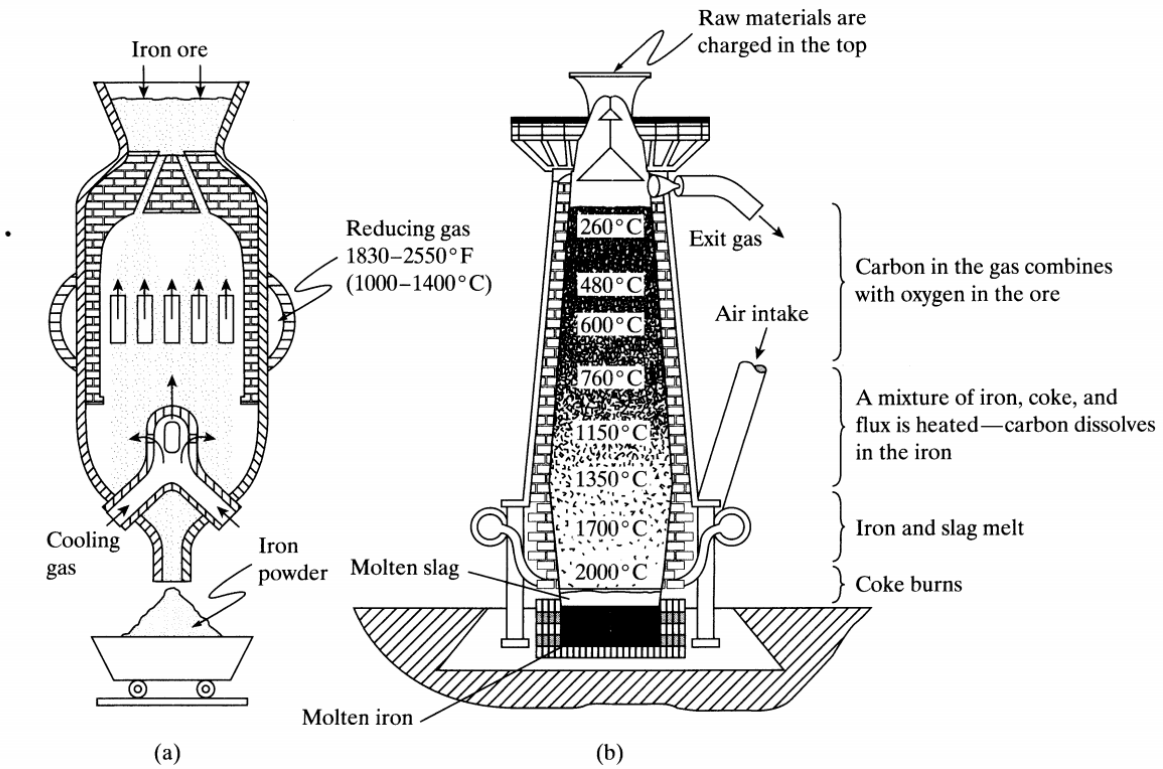


Figure 1 1: Schematic of a direct reduction process (a) and a blast furnace (b) [1]

There is various type of steel refining process available such as **Bessemer process**, **Open-hearth furnace**, **Basic Oxygen Furnace** (BOF), **Electric Arc Furnace** (EAF), **Secondary Steel making Process** and **Special Refining Process**. Open-hearth furnace process was invented in 1870 and by 1910 it had become the standard commercial process. Open-hearth furnace process reputation was overtaken by BOF and EAF in 1960 due to their overall batter performance. Nowadays, Approximately 60% of the production in US uses BOF, the remainder use EAF in refining Pig Iron.

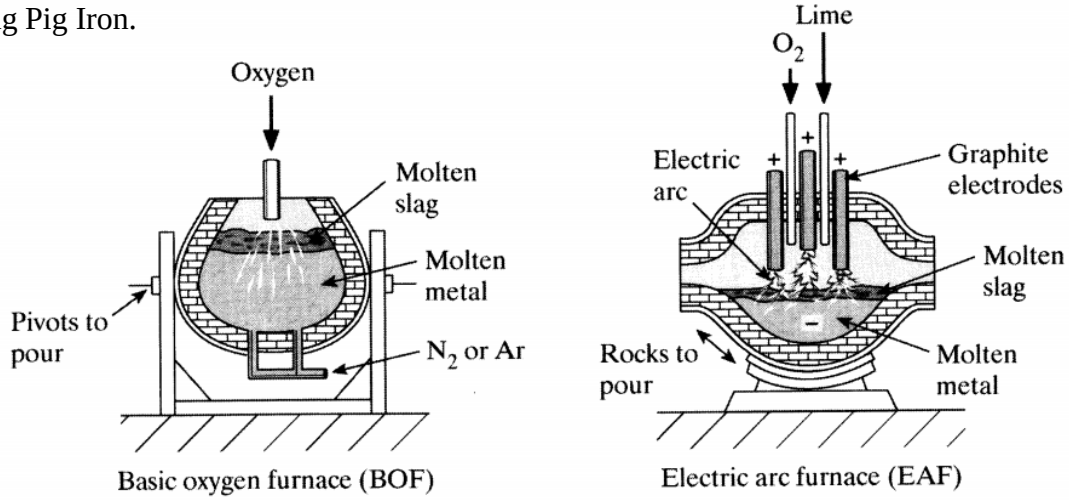


Figure 1 2: Schematic of Basic Oxygen Furnace and Electric Arc Furnace [1]

Special Refining Process subjected to special refining processes that are proposed to modify chemical composition and/or to remove impurities. This process consist of several sub process such as Vacuum arc remelting (VAR), Vacuum induction melting (VIM), Electron beam refining, and Electroslag melting (ESR). This process is suitable to produce Alloy steels, **Stainless Steels**, bearing steels, and tool steels. [1]

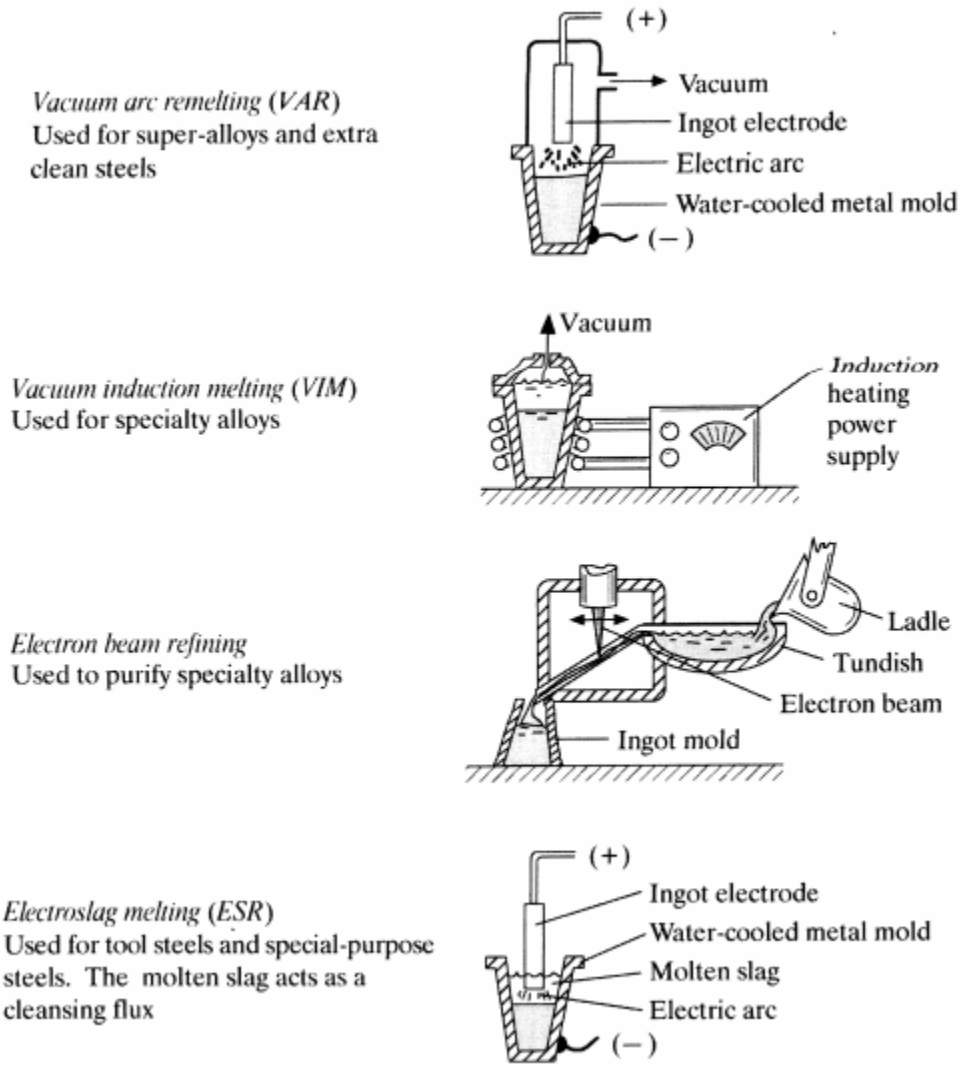


Figure 1 3: Special Refining Process [1]

1.1.2 Stainless Steels

Stainless steel is a type of steel which have sufficient corrosion resistant such as rust, easy for fabrication characteristic weld elements and also cast shapes. Generally stainless steel is a special alloy which contains iron, chromium, and any other elements with the intention of resist corrosion from many environments. Steels in order to be called as “Stainless”, the steels need to have certain amount of chromium at least around 10.5%. However, there are stainless steel having less than 10.5% chromium due to its insufficient amount of carbon. [1]

Stainless steel able to withstand corrosion due to the present of chromium elements inside the material, the chromium form a thin barrier around the grain boundaries to prevent oxidation happened. However chromium has high tendency to combine with carbon and oxygen. If the amount of carbon is too high, this will affect the structure of stainless steels. Oxidation also maybe happened if the manufacturer does not process it using special technique and process. [1] **Intergranular corrosion** is example of oxidation of chromium layer; happen due to the inadequate heat treatments, weldments and high-temperature service, which lead to formation of Carbide Precipitates around the grain boundaries. [2] To prevent this, stainless steel must avoid certain temperature (900℃ - 600℃) during the making process.

Stainless steel can be classified into several branches, which differentiated in variety ways such as in term of usage application, alloying elements, and also metallurgical phases present in their microscopic structures. **Ferritic Stainless steels**, **Austenitic Stainless Steels** and **Duplex Steels**. [3] [1] Ferritic stainless steels having BBC (Body-Center Cubic) microstructure, also having low carbon content around 0.19%, having 16% - 20% chromium content and also nickel free. Material such as Chromium, silicon, molybdenum, vanadium, aluminum, niobium, titanium and tungsten have higher tendency to help the formation of ferrite. [1]

Austenitic Stainless steel is more complex than ferritic stainless steels due to have 4 major alloying elements such as iron, chromium, carbon and nickel. The effect of nickel is to encourage a completely austenitic structure. Usually austenitic stainless steel comes with percentage of nickel at least 8% and can go high to 24%. In 8% to 10% nickel concentration range, the phase diagram show that austenite and ferrite are in equilibrium concentration. [1] Material such as Nickel, colbat, manganese, copper, carbon and nitrogen will facilitate the formation of Austenite. [1]

Duplex steels are combination of ferrite and also austenite elements in single alloy. These ferrite-austenite alloys were introduced in late 1970s with adjustments of ferritizers and austenitizers in same stainless steel allow the formation of structure having both ferrite and austenite. Duplex steel seem to have higher yield strength (twice all austenite alloy), having batter resistance to stress corrosion cracking and also enhanced weld ability. [3]

1.1.3 Heat Treatment

Heat treatment is process which used to improve the properties of the steels such as annealing, hardening; tempering depends on the properties desire. There a lot of type hardening process available such as Induction Hardening, Flame hardening, Laser and Electron Beam Hardening, and Diffusion Hardening. However, in this thesis we more focus on Diffusion hardening by using Nitriding method.

Nitriding process uses either Ammonia or Nitrogen in high temperature environment, resulting monatomic nitrogen diffuse into the surface of the steel being treated. [1] The reaction of the nitrogen with steel causes the formation of very hard iron and alloy nitrogen compound which sometimes even harder than any other tools and carburized steels. The advantages of this process is the hardness process can be achieved without other heat treating process such as air quenching, water quenching and many more. This process also can prevent scaling and discoloration on the surface as the hardening process is accomplished in a nitrogen atmosphere. [1] The diffusion rate of nitrogen in steels depends on the solubility temperature as well as other parameters such as nitriding time, and surface area.

1.2 Problem Statement

Stainless steel has high strength and high oxidation resistance due to the high chromium content between its grain boundaries. However, the problem arises on how to further improve the characteristic of the metal? Current heat treatment process such as carburizing and nitriding is enough to achieve the requirements of the material? Effects of low niriding process towards the microstructure of the steels? Properties of the material will improve proportionally with time of carburizing and nitriding?

1.3 Objective

The objectives of this project are to investigate the microstructure and micro hardness of nitrided AISI 430 Ferritic Stainless Steels.

1.4 Scope of the Study

(I) To characterize the microstructure of nitrided AISI 430 Ferritic Stainless Steels. Using Microscope, we study the characteristic of microstructure.

(II) To analyze the particle concentration and diffusion rate of nitrogen into AISI 430 Ferritic Stainless Steels. Using SEM and XRD analyzes the particle concentration different and diffusion rate to investigate the successful rate of the diffusion bonding.

(III) To Investigate the Time dependent towards the micro hardness of the AISI 430 Ferritic Stainless Steels and relate to the diffusion rate of nitrogen into the steels. By using Vickers hardness we measure the hardness at each different point.

1.5 Relevancy of study

(I) Few researches have been done on nitriding process to the Ferritic Stainless steel.

(II) AISI 430 Ferritic Stainless steel is easier to be manufacture due to its durability and soft compare to Martensitic Stainless Steels, Austenitic Stainless Steels and Duplex Stainless Steels.

1.6 Feasibility of Study

(I) Precise objective and scope of study

(II) All equipment need in completing the project available in Universiti Teknologi PETRONAS.

(III) Testing based on different perspective compare to previous research.

CHAPTER 2
LITERATURE REVIEW

2.1 AISI Ferritic Stainless Steels grade 430L

AISI 430 Ferritic Stainless steels are known as Stainless steel type 1.4016 in UK. This type of stainless steel has great properties such as good corrosion resistance, good formability and ductility. It also in pure form which is non-hardenable, and come with excellent finish quality. Having excellent resistance to nitric acid make it suit to be use for chemical applications. Non-hardenable make it easier to deform eventually make it popular in domestic appliances and decorative trim. [4]

Aside from corrosion resistance, AISI 430 Ferritic Stainless steel also having good heat resistance in term of oxidation, easily machined and weldability. Ferritic stainless steel can be ready welded by any fusion methods, however preheating to 150℃ to 200℃ is needed. The welded 430 FSS need to be annealing at 790℃ to 815℃ to reduce embrittlement of welded zone. [4]

Table 2 1: Chemical composition of AISI Ferritic Stainless Steels Grade 430 [5] [4]

Composition	Weight Percentage
Carbon, C	0.062
Chromium, Cr	15.72
Manganese, Mn	0.31
Molybdenum, Mo	0.13
Nickel, Ni	0.12
Sulphur, S	0.003
Silicon, Si	0.23
Phosphorus, P	0.03
Titanium, Ti	0.002
Iron, Fe	Balance

2.2 Introduction of Nitriding

Nitriding is a hardening technique to strengthen steels by diffusing nitrogen into surface of steels and iron. The solubility of Nitrogen depends on few factors such as **Furnace Temperature, Process control, Time, Gas flow, Gas activity control** and **Process chamber maintenance**. [6] Through experiment by Adolph Machlet, he discovers that Nitrogen was very soluble in iron due to it atomic small size compare to Iron. [7] [6] Nitrogen diffusion produced hard surface of the iron and significantly improve it corrosion resistance properties. [6]

There is few Nitriding process available, such as **The Floe Process, Salt Bath Nitriding, The ion, plasma nitriding** and **Gas Nitriding**. Floe Process carried out in two distinct events, the first eventis accomplished as normal nitriding cycle at temperature of 500℃ with 30% dissociation of ammonia. The first process will produce Nitrogen rich compound at the steel surface. Then the process will continue with second stage in which the temperature will be increase to 560℃ at 85% ammonia dissociation. The main function of second stage is to reduce formation of compound zone. [6]

Salt Bath Nitriding is develop right after the development of gas nitriding process, Unlike Gas nitriding and Floe process, this method uses molten salt as a nitrogen source. This process uses the principle of decomposition of cyanide to cyanide and independence of nitrogen within salt for the diffusion into steel surface. Meanwhile, Plasma nitriding uses plasma discharge of reaction gases to heat the steel as well as to supply nitrogen ions used for nitriding. [6]

Gas Nitriding is a simple technique which does not require any special equipment, this process involve nitriding at high temperature and nitriding at low temperature. High Temperature Gas Nitriding (HTGN) usually involves exposing the sample in nitrogen atmosphere in High temperature around 1000 -1200℃. Meanwhile, Low Temperature Gas Niriding (LTGN) involve exposing the sample in nitrogen environment at Low temperature around 500-600℃. [7]

During the heating process, iron particle receive energy and then vibrate even further each other, the vibration of the particle create spaces between the particle which eventually give space for nitrogen to enter and fill up the space between the particles. This nitrogen finally acts as dislocation barrier which will significantly increase the strength of the steels. Nitrogen gas is

optional in gas nitriding due to the Nitrogen properties which is easy to dissolve in metals and both non-toxic and not-explosive gas. [7]

After Nitriding Process, Compound zone or white layer created along the surface steels. [8] [6] After the Compound zone, there are few more zone available such as Diffusion zone, transition zone and core material. [6] White layer and diffusion zone is where diffusion occur rapidly and also where available a lot of metal compound precipitation.

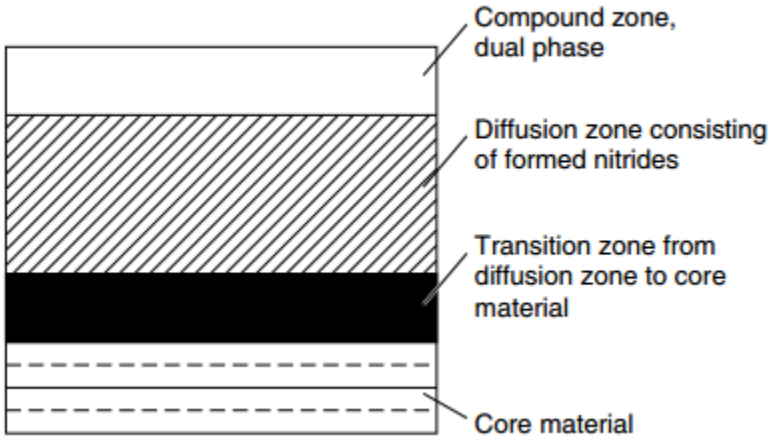


Figure 2 1: Schematic of a typical nitride case structure [6]

Strong attraction between Nitrogen ion and metal during Gas Nitriding, leads to formation of metal compounds such as Chromium Nitride and Ferrite Nitride. Refer to previous research, Precipitation of Metal-N nitrides usually occur in Low Temperature Gas Nitriding, while $Metal_2$-N were precipitated at High Temperature Gas Nitriding. [7] Formation of Chromium compound will reduce the corrosion resistance of metal due to depletion of Cr particle in the solid solution. [9]

High Temperature Gas Nitriding (HTGN) process which running at 1000-1200℃, higher than crystallizations temperature. The amount of nitrogen dissolve in the steel will influenced the microstructure of nitride steel. The microstructure probably transforms either to austenite or martensite depends on the amount of nitrogen diffused. [10] According to Nazirul Nazlan [11] in his research, phase changes of microstructure during nitriding caused by formation of nitride.

The concentration of nitrogen of LTGN is lower compare to HTGN, due to temperature different.

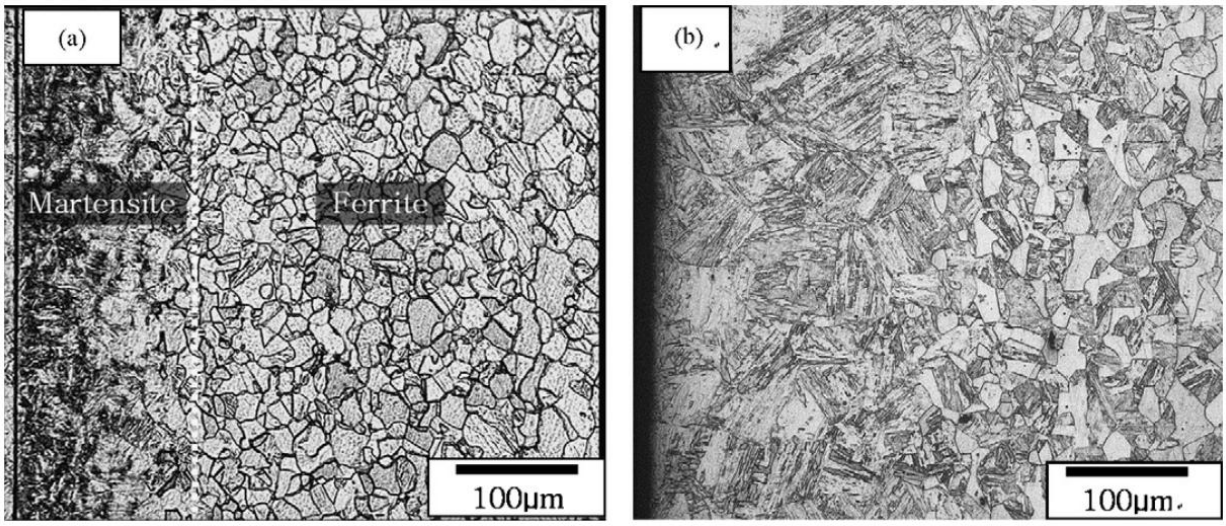


Figure 2 2: Optical micrograph of AISI 430 FSS after the HTGN treatment at 1100℃ (a) in 15 min and 1100℃ (b) in 10 hour [10]

Figure 2.2 (a) shows that the area near the surface was transform to martensite, while the interior area still remains as ferrite phase. Figure 2.2 (b) shows the depth of martensite phase increase further compare to Figure 2.2 (a). This show that by increasing the nitriding time will also increasing the penetration of nitrogen into steel. [10] Yet, the penetration of nitrogen depends on carbon content inside the steel. From previous research, that nitrogen did not permeate through the 0.13C-13 Cr martensitic stainless steel due to the effect of carbon. [10]

However, differ with Low Temperature Gas Nitriding (LTGN) process which running at 500-600℃, lower than crystallizations temperature. The amount of nitrogen dissolve in the steel probably will little bit influence the microstructure of nitride steel. Nevertheless, the more amount of nitrogen diffuse will enhance the strength of the steel due to increasing number of dislocation barrier available. [12]

2.3 Hardness Properties

Hardness Properties of the material can be done using Vickers pyramidal hardness test, this is an effective tool for characterizing the microstructure of materials according to ASTM E384-10 specification. Aside Vickers hardness, there also few other methods to measure the hardness properties of the material across a variety of load scale such as Brinell and Rockwell. Vickers Hardness value is measured by the ratio of Force F required to produce an indent with a given surface area A. [13] The H_V number is defined as the load divided by the surface area of indentation N/mm^2. [14]

Previous researches have been done toward HTGN, the corrosion resistance and hardness properties of the surface layer increase. However, there also some disadvantage of nitriding, aside increasing the surface hardness, it also increase the brittleness of the materials. [10] [7] The steel become harder due to increasing slip dislocation barrier; at the same time reduce the ductility of the metal since there limited dislocation spaces available, make it become brittle. [12]

Hassan [15] in his research state that, gas Nitriding process will enhance the hardness value at the surface, however the hardness value will drop to the centre of the samples due to the high nitrogen concentration at the surface area. However, due to limitation of diffusion and diffusion barrier, the concentration of nitrogen drops towards the centre of the samples.

CHAPTER 3
METHODOLOGY

3.1 Flow Chart

Below is the flow chart of this research. Start from selection of project title till the end by result presentation. Consist of 2 semester period project, 1st semester for FYP 1 and 2nd semester FYP2.

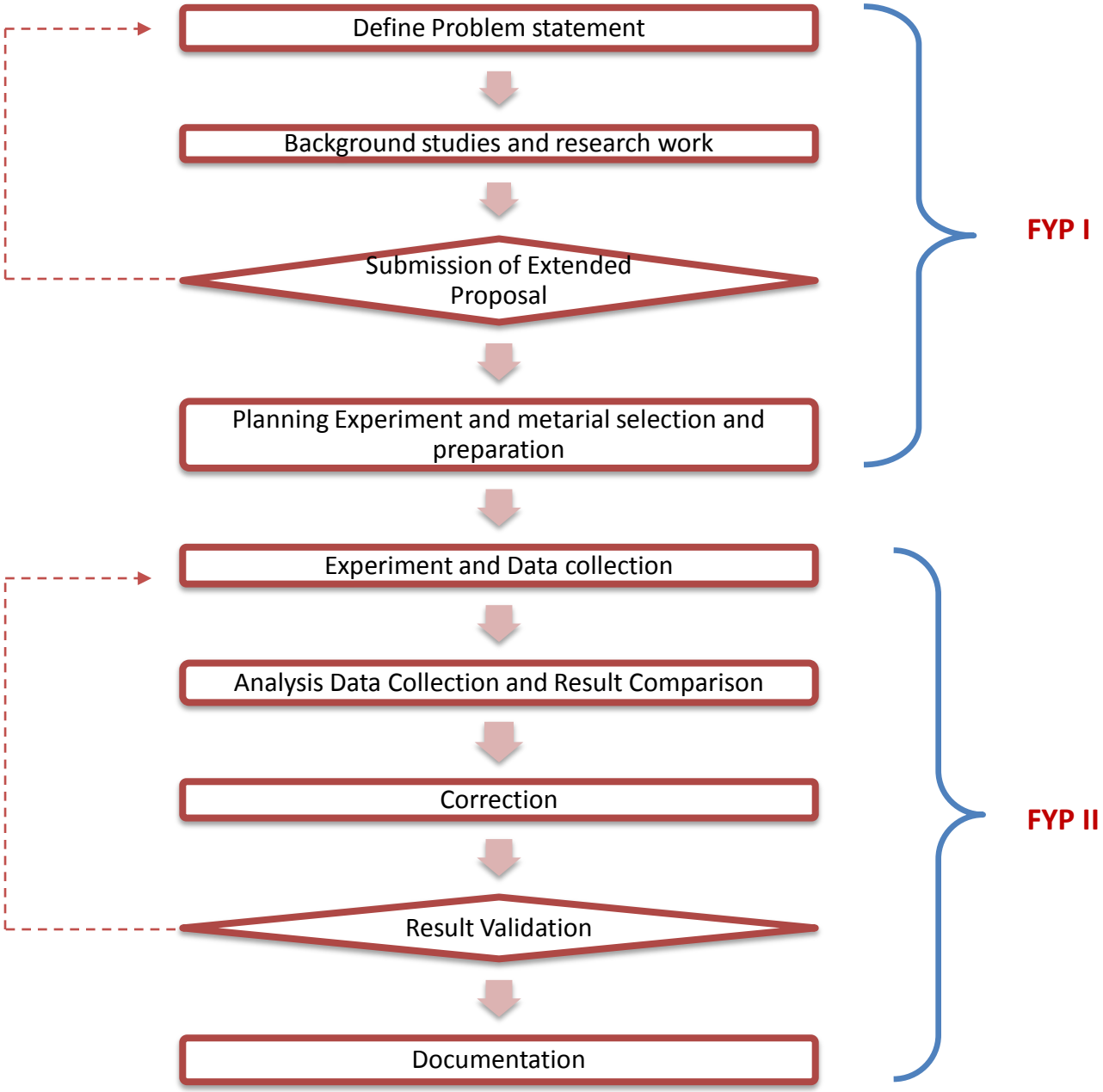


Figure 3 1: Flow Chart

3.2 GANTT CHART

Table 3 1: Gantt chart

Detail/Week		FYP I															FYP II												
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28
1	Topic Selection							MID SEM BREAK								SEM BREAK							MID SEM BREAK						
2	Preliminary Research Work																												
3	Submission of Extended Proposal									17/11/14																			
4	Proposal Defense																												
5	Project Work Continue - Preparation of experiment material - Further study of experiment expected result - Finishing Interim Report																												
6	Submission of Interim Draft Report													8/12/14															
7	Submission of Interim Report																												
8	Submission of Progress Repost																							20/3/15					
9	Conducting experiment: Polishing and nitriding												16/2/15																
10	Observe via optic microscope. Poster Presentation																												
11	Observe via SEM, EDAX																												
12	Analysis data and discussion, result																												
13	Dissertation and Technical Report Submission																								17/4/15				
14	Final Presentation																												20/4/15

3.3 Key Milestones

Key Milestone [FYP 1]

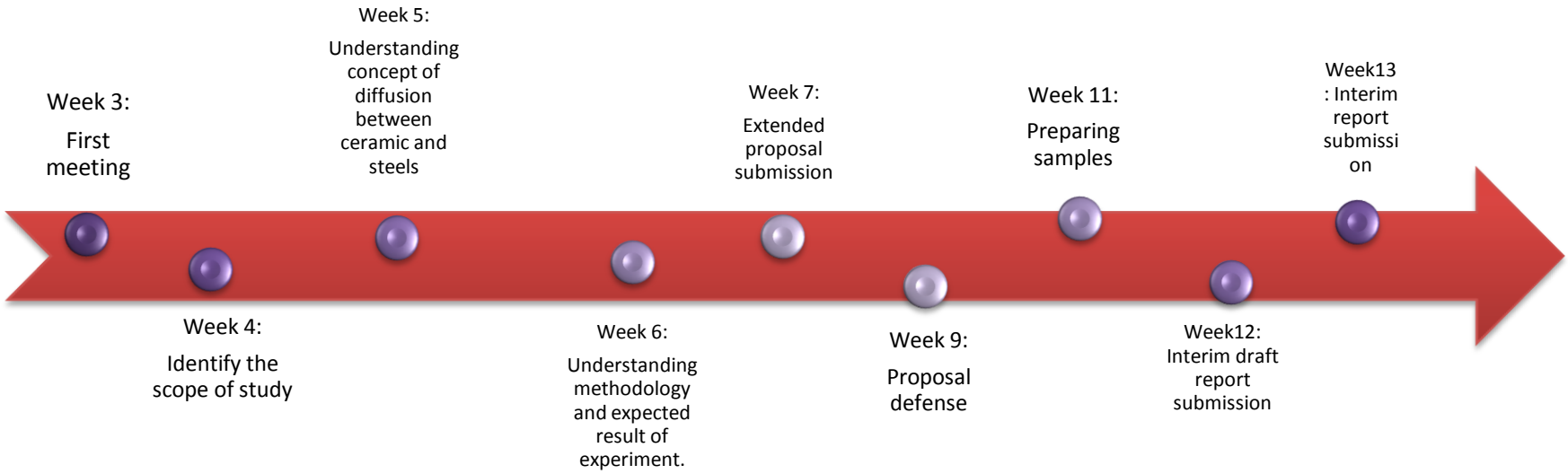


Figure 3 2: Key Milestone for FYP 1

Key Milestone [FYP 2]

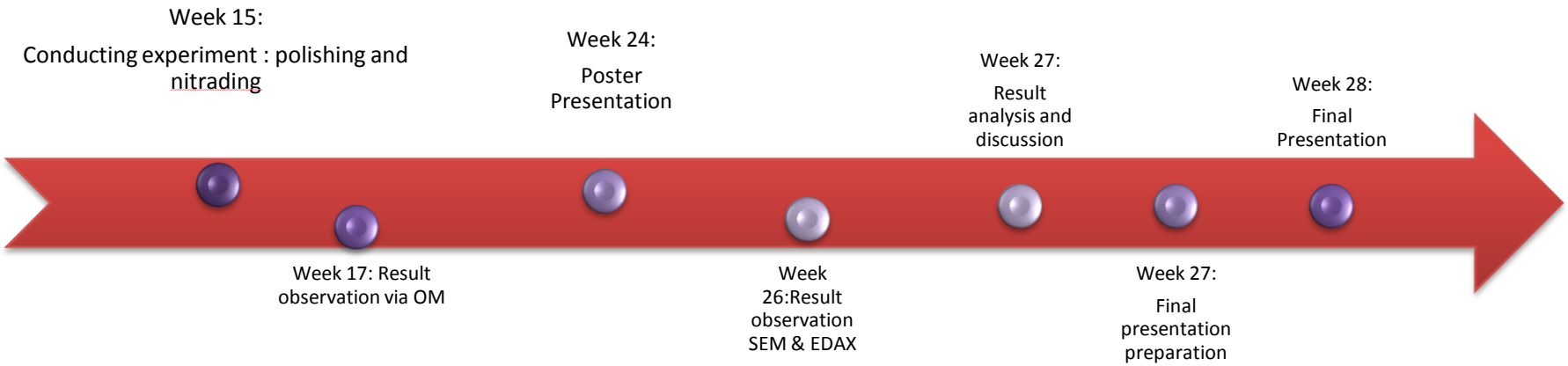


Figure 3 3: Key Milestone for FYP 2

3.4 Project Activities

This project having two major steps, first step is nitriding the sample, and second is observed the sample microstructure and phase diagram via microscope, EDAX and SEM.

3.4.1 Sample Preparation

Abrasive cutter, cutting sample

1. Power on the machine. Make sure light is on and fluid flow on the cutter disk.
2. Open the casing and place the sample and lock the sample to required position.
3. Close the casing and switch on the blade. Cut the specimen into 10mm long.
4. Switch off the blade along with open the casing. Unlock the sample and remove it.
5. Clean the sample and let it dry.
6. Repeat step 2-5 for other sample.
7. Switch off the machine and clean the workplace.

Samples Mounting

1. Since the samples are too small, the sample need mount in order to make the polishing process easier.
2. Use sample with diameter of 10mm and length of 10mm
3. Switch on the mounting machine
4. Clean the mould to remove and residue of leftover.
5. Place the surface of the sample that is to be tested. Insert suitable amount powder into the sample casing and lock the casing.
6. Parameter of mounting: Pressure – 4000psi, Heating time – 3 minutes, Cooling time – 2 minutes
7. Press start cycle button on the mounting machine.
8. Machine will "beep" when it is finish. Take out the finished mount.
9. Repeat step 2 – 6 for other samples.

Switch off the mounting machine and clean the workplace

Sample grinding and polishing

1. Install 400 grade sand papers on the grinding machine. Allow water to flow on the sand paper during grinding.
2. Switch on the grinding machine and grind the mount sample until it has mirror like finish.
3. Change the grade sand paper to grade 800, 1200, 1500, and 2500 depends on the type of materials. (**from low to high**)
4. Repeat step 1 -3 for other example.
5. Spray liquid polisher on the polisher and on the surface of the sample.

6. Polish until it get free scratch or little scratch on sample surface.
7. Proceed to final polishing using semi-liquid diamond polisher.
8. Clean the sample surface and apply ethanol on the surface to remove water from the surface of the sample.
9. Repeat step 5 – 8 for other sample. Clean the work place once finish
10. Remove the mount by using cutting tools. (Precaution: prevent any damaged to polished surface.)

3.4.2 Nitriding

1. Take samples to be nitriding
2. Clean the sample via cotton or cloth.
3. Connect all power sources to the furnace. Check the gas flow and make sure the furnace work properly as shown in **Figure 7.4**.
4. Parameters of furnace:
   Temperature: 600℃, Time taken: 2 to 24 hours, Pressure: 50 to 100mmHg, Gas composition: $500cm^3min^{-1}$ N_2 and $500cm^3min^{-1}NH_3$.
5. Put the sample on a mold provided and put it inside the furnace.
6. Use single-staged nitriding process. Temperature is set to 500℃ for low temperature nitriding and 1200℃ for high temperature nitriding.
7. Close furnace and start flow of ammonia gas at fast flow rate.
8. Set furnace temperature control at 150℃ simultaneously. Heat furnace to this temperature but do not exceed.
9. When the furnace has been purged to the degree that 10% or less air and 90% or more ammonia are present in the retort, the furnace may be heated to the nitriding temperature 600℃.
10. Maintain this rate for 4 to 10 hours. Important: nitriding cycle begin with a dissociation rate of about 15% to 35%.
11. Once finish, leave the samples inside the furnace, for slow cooling process.
12. Set up the same parameters for another sample.

Quench to prevent **carbide precipitation** formation which lead to intergranullar corrosion. (Avoid 900℃ − 600℃)

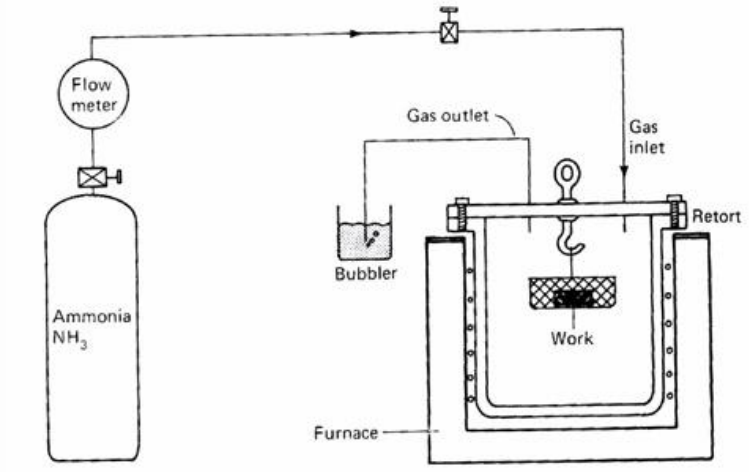


Figure 3 4: Schematic Diagram of Furnace Gas Nitriding [20]

3.4.3 Mounting Sample

1. Use sample with diameter of 10mm and length of 10mm
2. Switch on the mounting machine
3. Clean the mould to remove and residue of leftover.
4. Place the surface of the sample that is to be tested. Insert suitable amount powder into the sample casing and lock the casing.
5. Parameter of mounting: Pressure – 4000psi, Heating time – 3 minutes, Cooling time – 2 minutes
6. Press start cycle button on the mounting machine.
7. Machine will "beep" when it is finish. Take out the finished mount.
8. Repeat step 2 – 6 for other samples.
9. Switch off the mounting machine and clean the workplace.

3.4.4 Sample grinding and polishing

1. Install 400 grade sand papers on the grinding machine. Allow water to flow on the sand paper during grinding.
2. Switch on the grinding machine and grind the mount sample until it has mirror like finish.
3. Change the grade sand paper to grade 800, 1200, 1500, and 2500 depends on the type of materials.
4. Repeat step 1 -3 for other example.
5. Spray liquid polisher on the polisher and on the surface of the sample.
6. Polish until it get free scratch or little scratch on sample surface.
7. Proceed to final polishing using semi-liquid diamond polisher.
8. Clean the sample surface and apply ethanol on the surface to remove water from the surface of the sample.
9. Repeat step 5 – 8 for other sample. Clean the work places once finish

3.4.5 Etchant preparation, Viella's Etching [16]

1. Switch on the fume hood. All experiment involving chemicals must be prepared in this fume.
2. Prepare a clean beaker, two 100ml test tube and a stirrer.
3. Weight 1 gram of Picric acid ($C_6H_3N_3O_7$), 4ml Hydrochloric acid (HCL) and 96ml ethanol.
4. Mix 1 gram of Picric acid (in powder form) and 4ml Hydrochloric acid into the beaker and stir until it completely dissolves.
5. Once Picric acid is completely dissolves, add 96ml ethanol and stirrup the mixture until it completely mix up.
6. Viella's Etching is ready.

3.4.6 Etching sample

1. Use sample that has been polished. All steps are done in fume hood and use gloves for protection
2. Apply ethanol on the sample surface and put it on the heater. Then Let it dry.
3. Apply Viella's solution on the surface of the sample by swabbing using cotton. When the surfaces appear dull, stop swabbing and clean the surface using water.
4. Apply ethanol on the surface and put it on heater. Then let it dry.
5. Repeat step 1 – 4 for other sample. The sample is ready for next steps.
6. Clean the workplace, Gloves and cotton are put in the special dustbin as well as chemical waste.

3.4.7 Optical microscope

1. Power on the optical microscope and the computer; make sure all the cables connected properly as shown in **Figure 7.3**.
2. Place the etched specimen on the microscope.
3. By using 500 times magnifying glass, focus the image and capture the image and save it on the computer.
4. Use 100 times magnifying glass, focus the image and capture the image and save it on the computer.
5. Repeat step 4 – 5 for other specimen.
6. Power off the microscope and the computer.
7. The image taken is ready to be analyzed and studied.

3.4.8 Vickers Hardness Test

(ASTM E384: Standard Test Method for Micro indentation Hardness of Materials) [13]

1. Polish the surface of Nitrided Ferritic Stainless Steel samples by using sand paper
2. Vickers Hardness testing using LECO LM247 AT micro hardness tester as shown in **Figure 3.5 and 3.6**.
3. Turn on the machine, and place the specimen on the specimen table.
4. Vickers testing using procedure 11 surface positions on each specimen to carry out the hardness test.
5. Set the Force value to 100 gf (Hv 100), adjust the focus until the surface can be see clearly via microscope.
6. Press "start", the indenter is focusing to press into the specimen.
7. Keep for 10 to 15 seconds (dwell time) until the indenter is completed
8. Reading the horizontal and vertical indenter length and recorded the hardness reading in table.
9. Step 1 to 8 is repeated for other specimens.
10. Take the average reading Vickers Hardness.

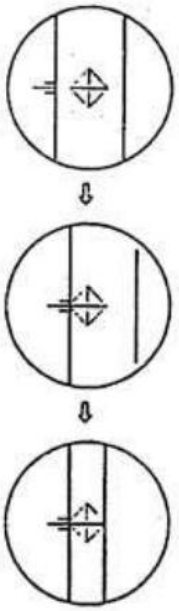


Figure 3 5: Shows the step on measuring an indentation. [13]

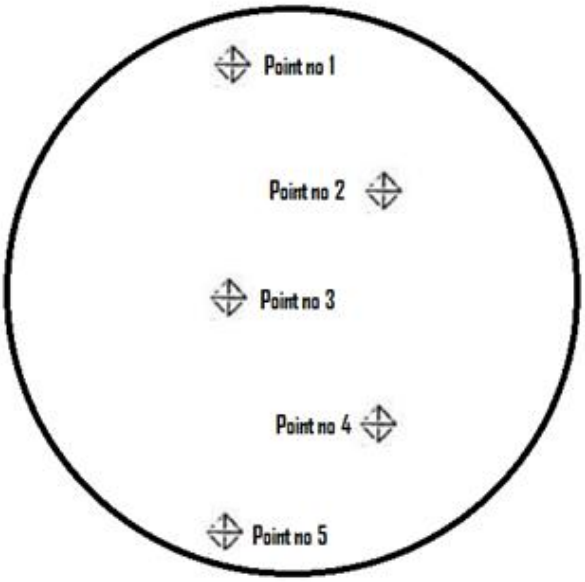


Figure 3 6: Shows the indentation location for overall Micro hardness Properties of FSS

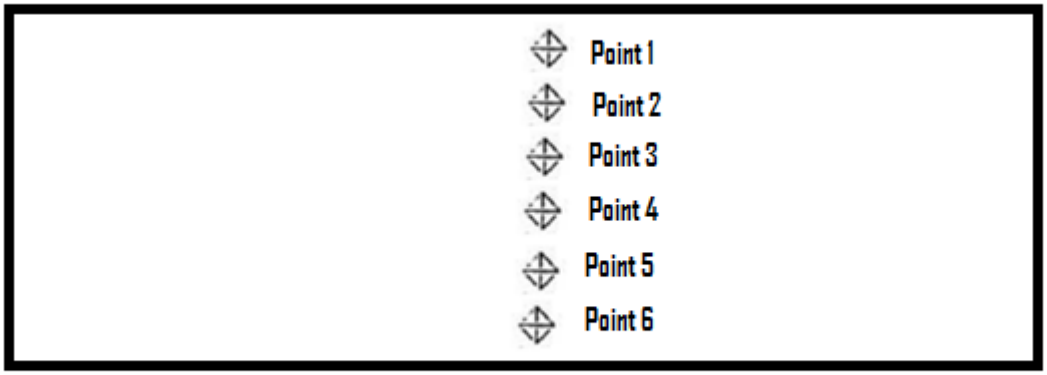


Figure 3 7: Shows indentation location for Cross section micro hardness properties of FSS

Table 3 2: Sample Data of Vickers Hardness Test

Nitriding Hours	**Argon -2 Hours**	**2 Hours**	**8 Hours**	**24 Hours**
Vickers Hardness				
1				
2				
3				
4				
5				
6				
7				
8				
9				

3.4.9 SEM and EDX

1. Observing the microstructure and element composition SEM and EDAX by using Scanning Electron Microscope (SEM) Phenom Pro-X. [17] Using data from **Figure 7.1**, it analyzes the particle dispersion of every element.
2. Few precautions before using Scanning Electron Microscope Phenom Pro-X, as shown in **Figure 7.5** and **Figure 7.6**.
   I. 1st Sample need to be dry.
   II. 2nd Sample must be firmly attached to sample stub/holder.
   III. For Charging Samples, Charge Reduction Sample Holder can be used to minimize charging effects.
   IV. Never prepare a sample in the Phanom sample holder to prevent contaminate the SEM Column.
3. Place the sample in the holder
4. Adjust the holder height; make sure the sample is same level with the sample holder.
5. Please Rotate the Holder height another 180°, to make sure it is exactly suitable with the height
6. Place the Holder in the Phanom Pro-X
7. Open up the software and the sample is ready to be analyzed.

3.5 TOOLS FOR DEVELOPMENT

Below are the tools needed, upon completion of this project.

Table 3 3: Tools and Software being used in this project

Elements	**Software/Platform**
Basic Tools	Microsoft Word, Microsoft Excel
Engineering Tools	Microscope, polishing machine, EDAX, SEM, Furnace Nitriding
Website Platform	UTPedia, MyAthens, Sciencedirect

CHAPTER 4
RESULT AND DISCUSSIONS

4.1 Optical Microscope

Figure 4.1 and Figure 4.2 show the microstructure of Unnitrided FSS at 100x magnification and 500x magnification respectively. Figure 4.3 and Figure 4.4 show the microstructure of Nitrided FSS in Ammonia environment for 2 Hours at 600℃ at 100x Magnification and 500 x magnifications correspondingly. 100x and 500x magnifications in Figure 4.5 and Figure 4.6 are of microstructure of Nitrided FSS in Ammonia environment for 8 Hours at 600℃. Meanwhile, Figure 4.7 and Figure 4.8 show the microstructure of Nitrided FSS in Ammonia environment for 24 Hours at 600℃ at magnification of 100x and 500x accordingly. All samples are under slow cooling process.

Figure 4.2 shows that the sizes of grain boundaries are bigger compare to the rest of the sample, this due to the slow cooling process during heat treatment and manufacturing process at high temperature (1000℃ to 1200℃) which higher than crystalline temperature. Throughout the cooling process, the small grain will merge each other resulting bigger size of grain formed.

Figure 4.3, Figure 4.5 and Figure 4.8 show that the number of grain boundaries increases with nitriding time, the size of grain boundaries decreases with nitriding time. The Metal-Nitride Precipitation concentration increases as shown in Figure 4.4, Figure 4.6 and Figure 4.7 respectively, this conclude that increasing in nitriding time will increase hardness of material at the same time also increase the Metal-Nitride concentration in the steels.

4.1.1 Unnitrided Ferritic Stainless Steel

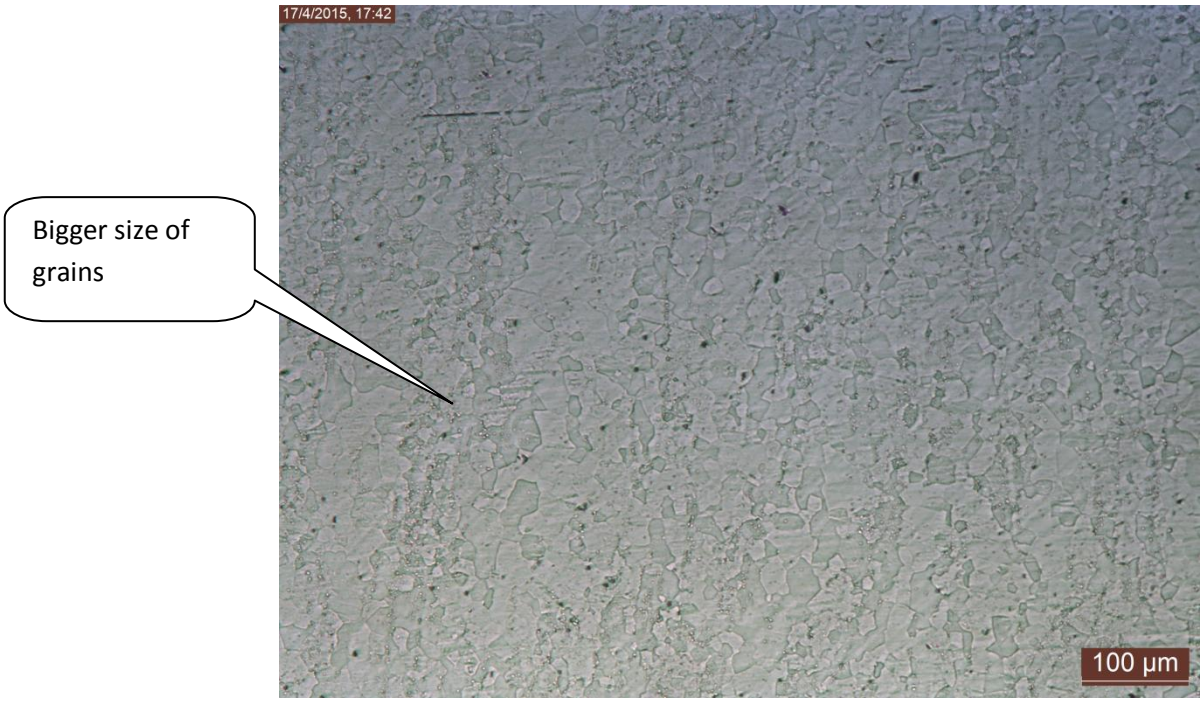


Figure 4 1: Optical Microscope of Unnitrided FSS (100x Magnification)

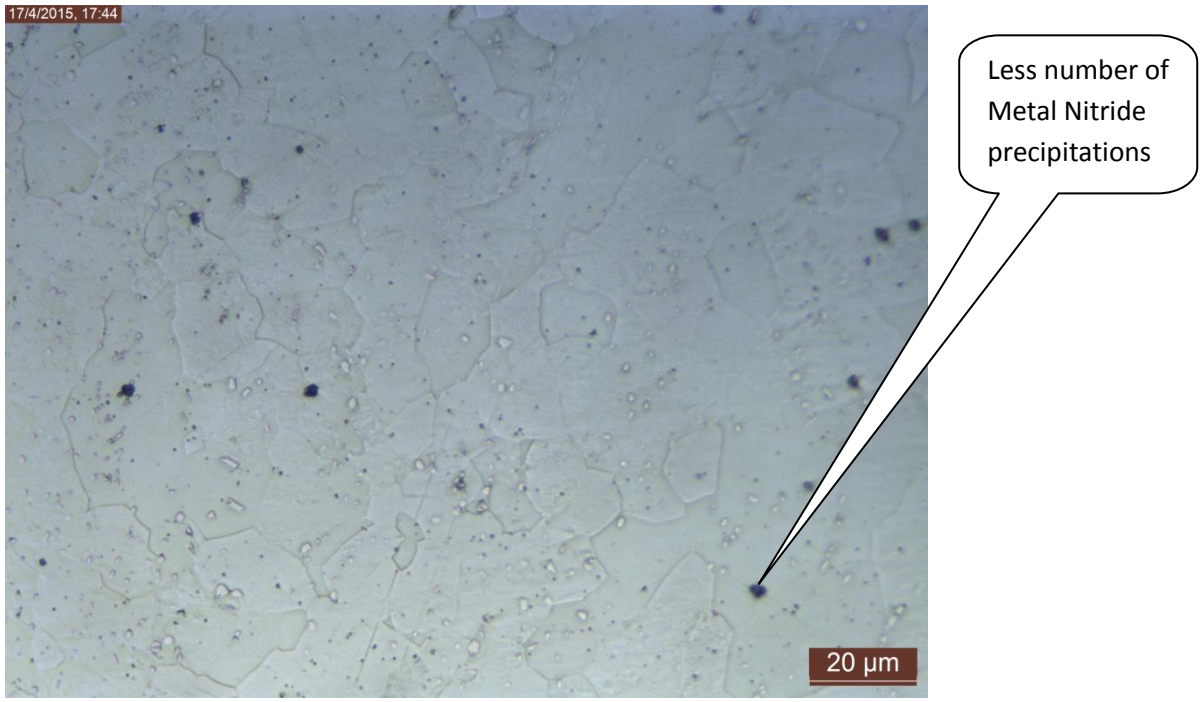


Figure 4 2: Optical Microscope of Unnitrided FSS (500x Magnification)

4.1.2 Nitrided Ferritic Stainless Steel for 2 hours in Ammonia Environment at 600 ℃ (Slow cooling)

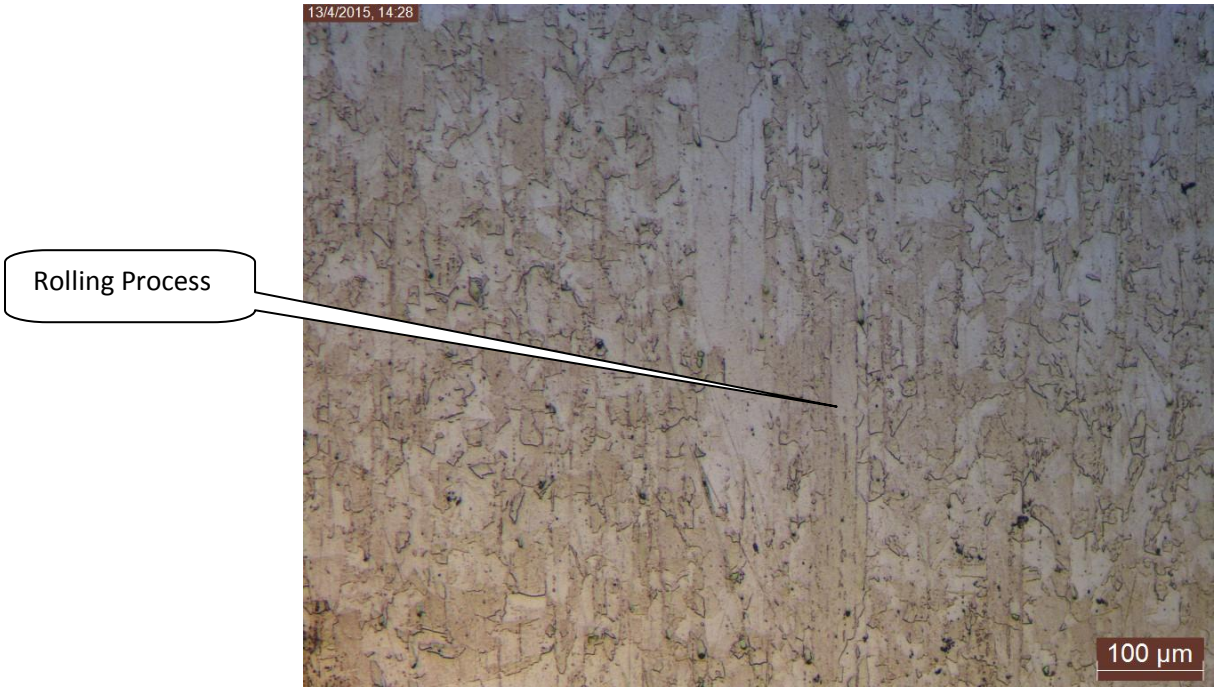


Figure 4 3: Optical Microscope of Nitrided FSS for 2 Hours in Ammonia Environment at 600 ℃ (100x Magnification)

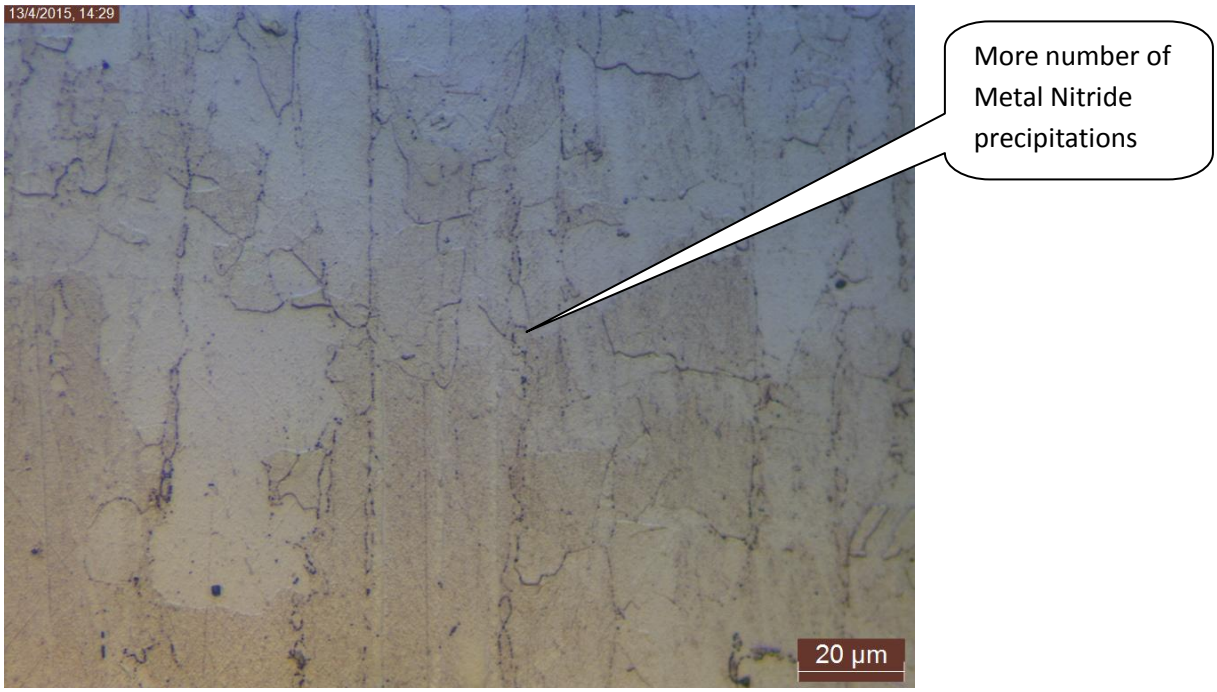


Figure 4 4: Optical Microscope of Nitrided FSS for 2 Hours in Ammonia Environment at 600℃ (500x Magnification)

4.1.3 Nitrided Ferritic Stainless Steel for 8 hours in Ammonia Environment at 600 ℃ (Slow cooling)

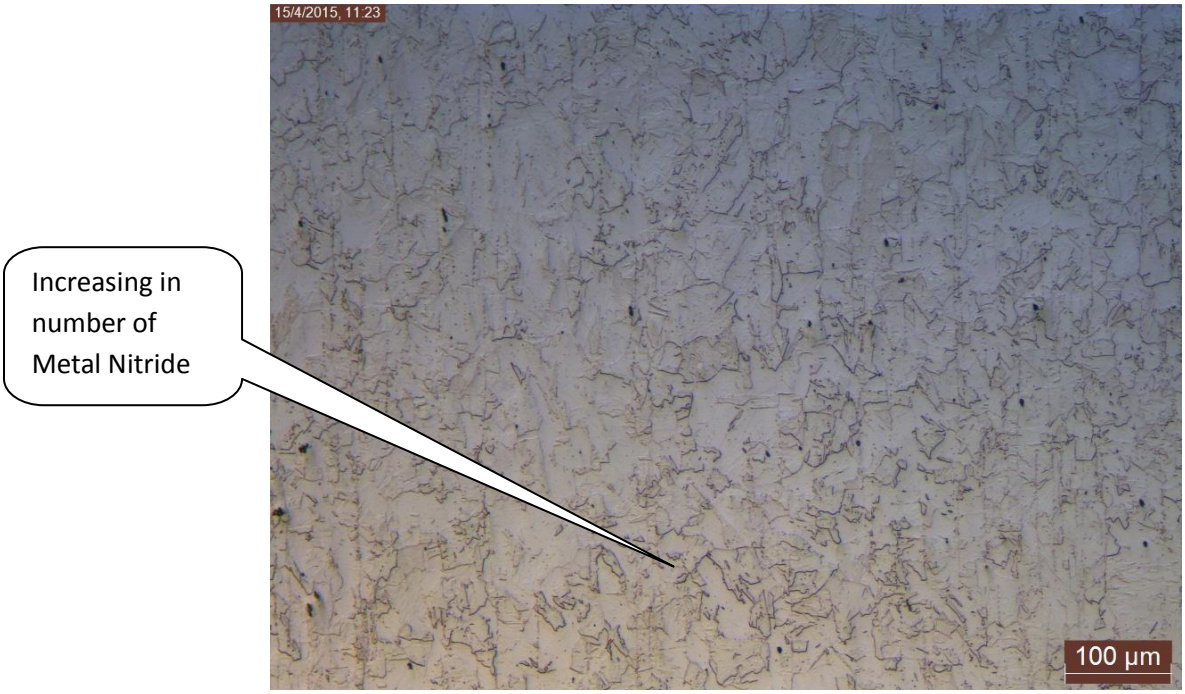


Figure 4 5: Optical Microscope of Nitrided FSS for 8 Hours in Ammonia Environment at 600℃ (100x Magnification)

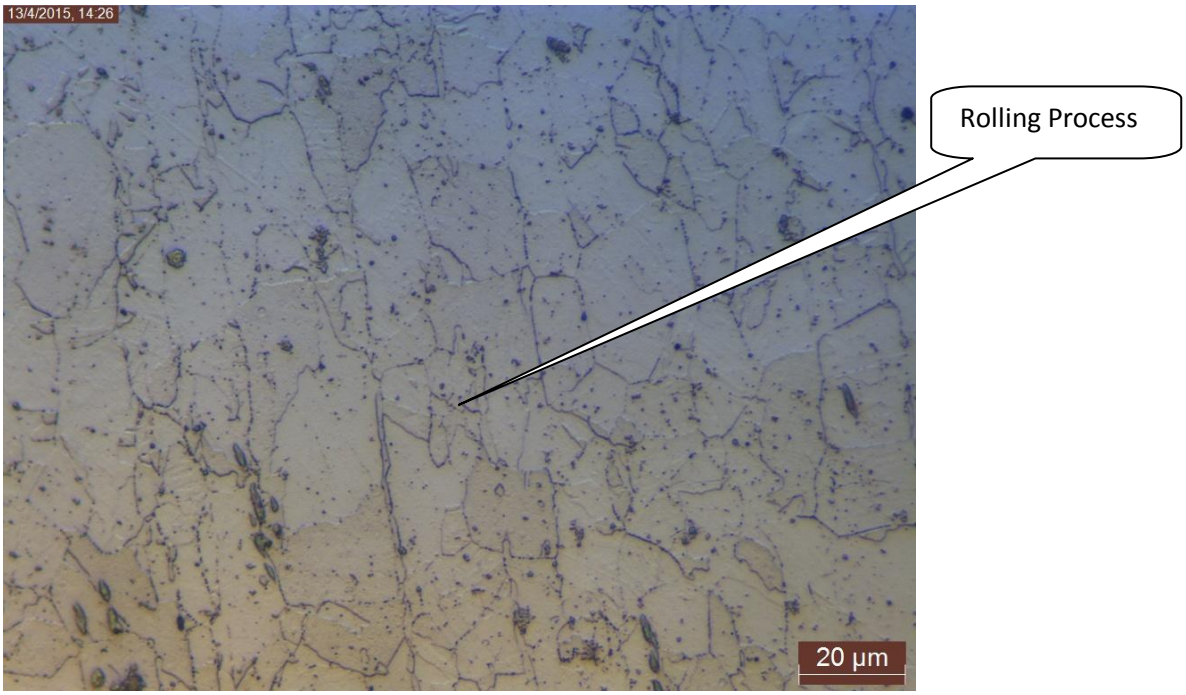


Figure 4 6: Optical Microscope of Nitrided FSS for 8 Hours in Ammonia Environment at 600℃ (500x Magnification)

4.1.4 Nitrided Ferritic Stainless Steel for 24 hours in Ammonia Environment at 600 ℃ (Slow cooling)

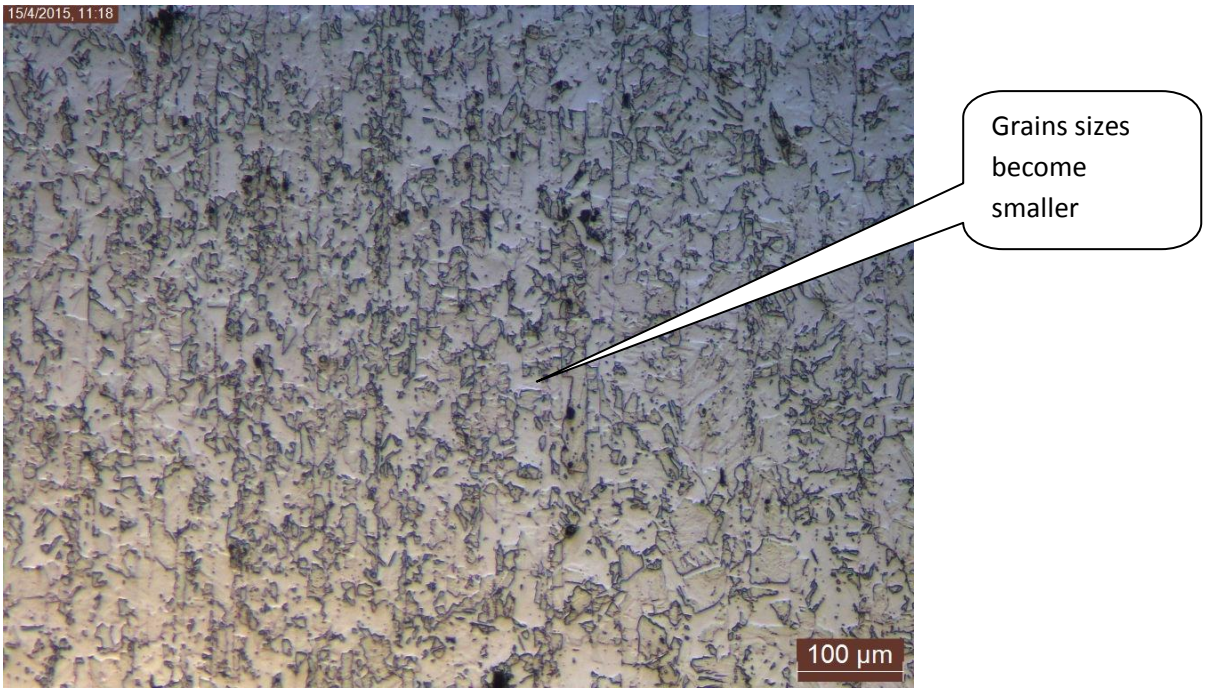


Figure 4 7: Optical Microscope of Nitrided FSS for 24 Hours in Ammonia Environment at 600℃ (100x Magnification)

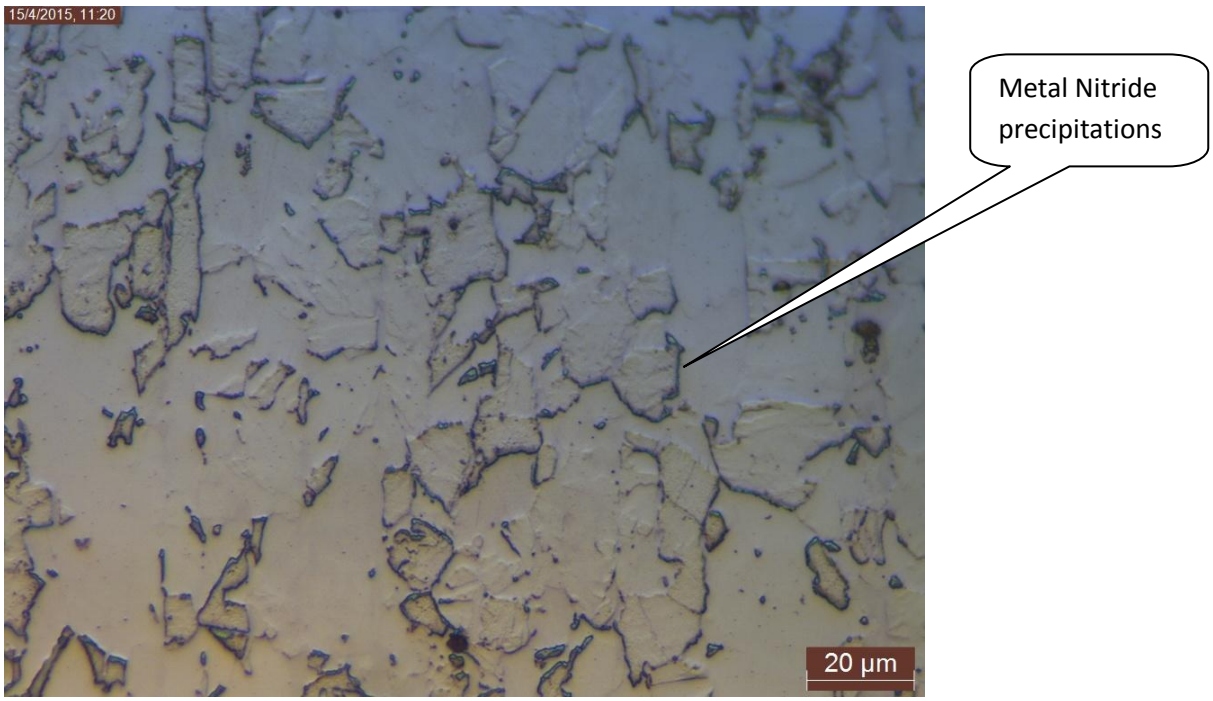


Figure 4 8: Optical Microscope of Nitrided FSS for 24 Hours in Ammonia Environment at 600℃ (500x Magnification)

4.2 Micro Hardness Properties

4.2.1 Overall Area Micro hardness properties

Vickers Hardness testing conducted to investigate hardness at random area of the samples. The indentation locations have been show in Figure 3.4 above, with spacing around 20μm between and Zip-Z pattern of indentation. The Vickers Hardness testing conducted using 100gf load, equivalent to 100 Hv. The Result of Vickers Hardness of overall area as shown in Table 4.1 and Figure 4.9 below.

Table 4.1 and Figure 4.9 shows that the comparison Vickers hardness for Unnitrided FSS, Nitrided FSS in Ammonia environment for 2 hours, 8 hours and 24 hours. From the result shows that the hardness of FSS increases with Time Nitriding, Unnitrided FSS have lowest hardness due to High Temperature heat treatment (1000-1200℃) which above crystalline temperature follows by slow cooling, lead to merging of grains, forming bigger size grain, reducing the number of grain boundaries available, thus reducing strength of material.

Table 4 1: Vickers hardness value for overall cross section FSS

Nitriding Hours	Unnitrided	2 Hours	8 Hours	24 Hours
1	160	180.8	215.8	241.2
2	163.2	176.8	215.6	241.1
3	162.5	191.2	246.3	229.2
4	183.5	175.1	207	206.3
5	163.2	171.5	207.9	206.1
6	164.3	186.6	229.3	210.6
7	164	180.3	238.9	224.9
8	159.4	194	231.9	240.9
9	170.9	187.1	229.5	236.8
Average Vickers Hardness	165.667	182.600	224.689	226.344

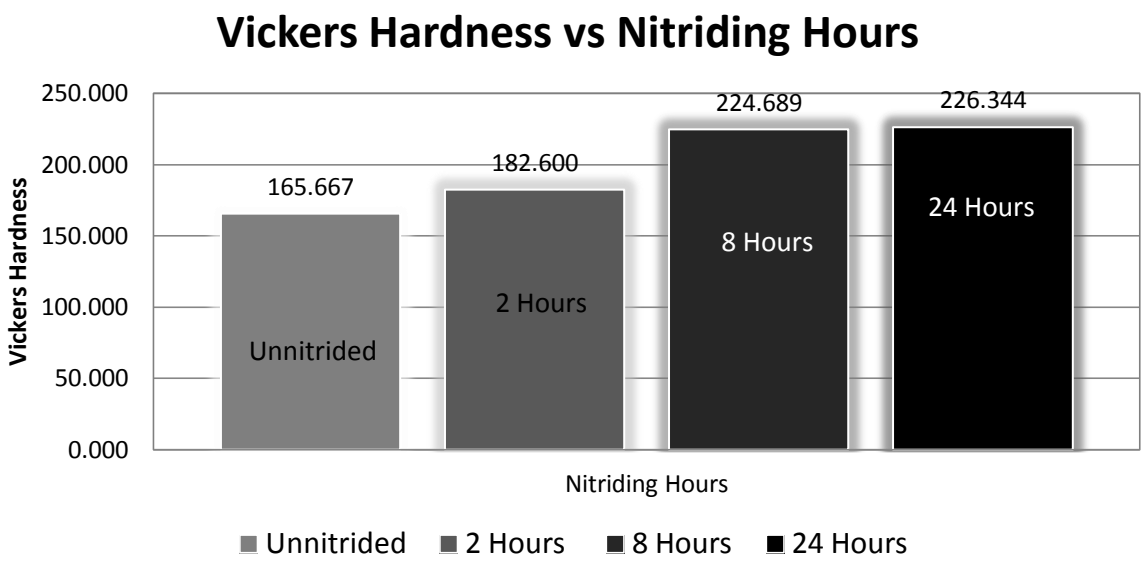


Figure 4 9: Vickers Hardness value for overall cross section FSS

4.2.2 Cross- section Micro hardness Properties

Figure 4.10 shows the Vickers Hardness value with the depth under the surface for Unnitrided and Nitrided Ferritic Stainless steel, Unnitrided FSS, Nitrided in Ammonia environment for 2 hours, 8 hours and 24 hours. For Unnitrided FSS, the hardness value changes randomly with the depth under the surfaces. Meanwhile, for Nitrided FSS in Ammonia environment, the hardness value decreases with increases of depth under the surface, proportional to the nitrogen content diffuse under the surface of Nitrided FSS for 2 hours, 8 hours and 24 hours.

The surface having high hardness value due to the high concentration of nitrogen available compare to Inner area due to diffusion barrier and diffusion limitation to the centre of the materials. Vickers hardness of unnitrided FSS change randomly with cross section distance due to unbalance cooling of the material and different concentration of inert particle inside the material. Outer layer will cool faster than inner layer resulting formation of bigger size of grains in the inner area.

Table 4 2: Cross Section surface hardness of Unnitrided FSS

Unnitrided				
Indentation Depth (µm)	Vickers Hardness (Hv)			
	Point 1	Point 2	Point 3	Average
80.00	162.00	154.80	150.90	155.90
160.00	168.70	163.20	154.20	162.03
240.00	154.30	177.30	162.50	164.70
320.00	175.80	183.50	160.80	173.37
400.00	163.20	175.50	172.90	170.53
480.00	163.30	164.30	176.20	167.93
560.00	167.30	150.80	164.00	160.70
640.00	153.20	159.40	153.90	155.50
720.00	170.90	170.40	171.30	170.87

Table 4 3: Cross section surface hardness of Nitrided FSS in Nitrogen Environment for 2 Hours

Nitrided in Nitrogen for 2 Hours follow by Slow cooling				
Indentation Depth (µm)	Vickers Hardness (Hv)			
	Point 1	Point 2	Point 3	Average
80.00	180.80	193.30	193.20	189.10
160.00	178.00	176.80	188.40	181.07
240.00	166.50	186.00	191.20	181.23
320.00	153.30	175.10	182.10	170.17
400.00	171.50	177.90	178.60	176.00
480.00	173.90	186.60	188.20	182.90
560.00	170.20	189.90	180.30	180.13
640.00	176.00	194.00	190.90	186.97
720.00	187.10	206.50	199.20	197.60

Table 4 4: Cross section surface hardness of Nitrided FSS in Nitrogen Environment for 8 Hours

Nitrided in Nitrogen for 8 Hours follow by Slow Cooling				
Indentation Depth (µm)	Vickers Hardness (Hv)			
	Point 1	Point 2	Point 3	Average
80.00	215.80	241.70	255.10	237.53
160.00	211.80	215.60	254.10	227.17
240.00	211.70	216.70	246.30	224.90
320.00	208.70	207.00	249.30	221.67
400.00	207.90	214.80	223.30	215.33
480.00	222.30	229.30	226.90	226.17
560.00	225.60	232.30	238.90	232.27
640.00	230.00	231.90	244.60	235.50
720.00	229.50	229.60	247.50	235.53

Table 4 5: Cross section surface hardness of Nitrided FSS in Nitrogen Environment for 24 Hours

Nitrided in Nitrogen for 24 Hours follow by Slow cooling				
Indentation Depth (µm)	Vickers Hardness			
	Point 1	Point 2	Point 3	Average
80.00	231.00	241.20	230.70	234.30
160.00	225.50	241.10	233.00	233.20
240.00	211.60	218.30	229.20	219.70
320.00	215.10	206.30	220.30	213.90
400.00	205.70	206.10	203.40	205.07
480.00	187.00	210.60	194.10	197.23
560.00	218.80	211.60	224.90	218.43
640.00	226.20	240.90	222.60	229.90
720.00	228.10	236.80	230.40	231.77

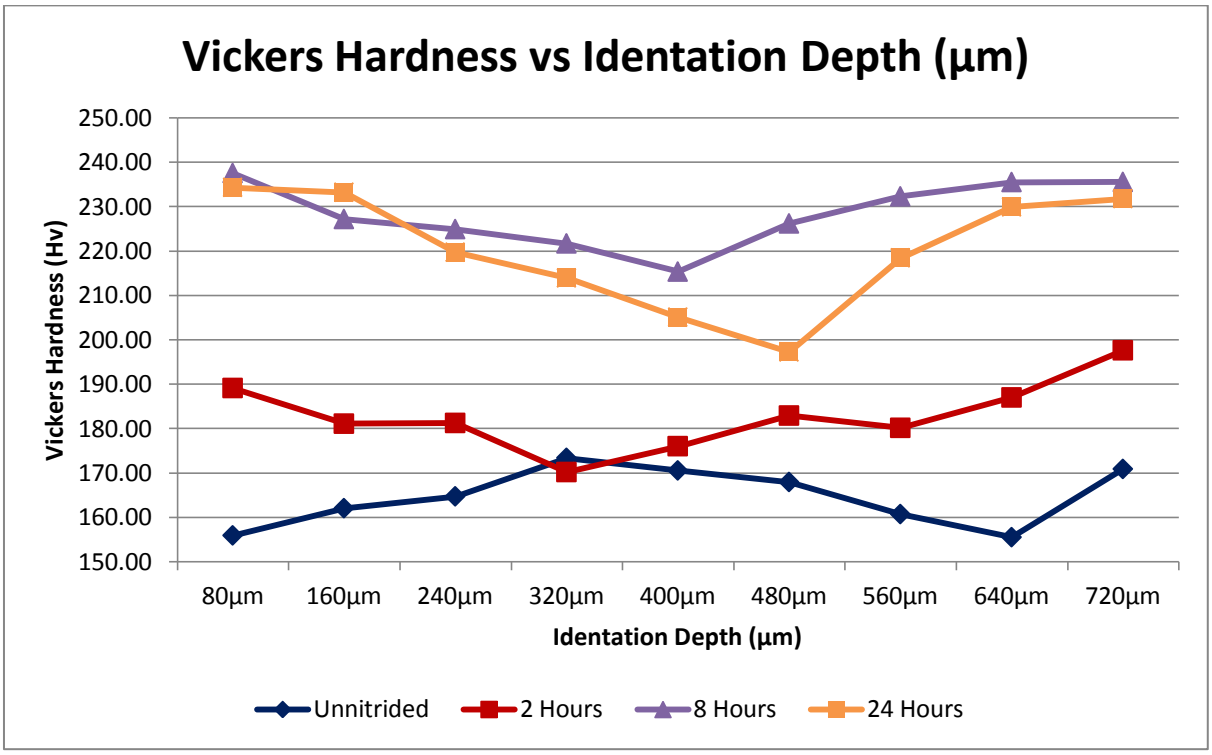


Figure 4 10: Cross-section hardness of Nitrided Ferritic Stainless Steels

4.3 Scanning Electron Microscope EDAX

4.3.1 Unnitrided Ferritic Stainless steel

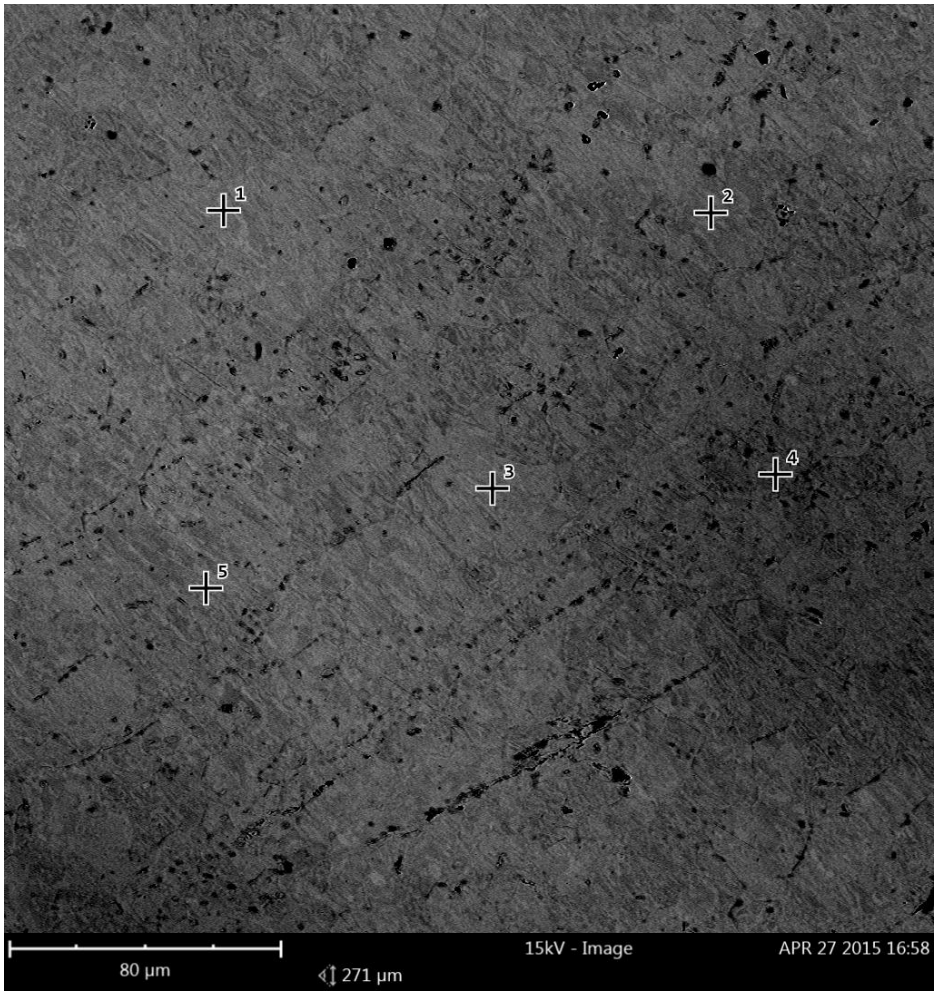


Figure 4 11: SEM- EDX of Unnitrided FSS.

Table 4 6: Chemical Composition of Unnitrided FSS.

Point		**1**	**2**	**3**	**4**	**5**
Chemical Composition (wt %)	Iron (Fe)	80.4	81.0	81.1	83.1	81
	Nitrogen (N)	0.0	0.0	0.0	0.0	0.0
	Chromium (Cr)	16.5	16.1	16.7	15.6	16.2
	Manganese (Mn)	0.7	0.8	0.7	0.3	1.0
	Molybdenum (Mo)	0.9	0.8	0.3	0.2	1.0
	Nickel (Ni)	0.6	0.5	0.5	0.0	0.0
	Silicon (Si)	0.6	0.4	0.5	0.5	0.5
	Phosphorus (P)	0.3	0.3	0.2	0.2	0.3
	Titanium (Ti)	0.0	0.1	0.0	0.0	0.0
	Sulphur (S)	0.0	0.0	0.0	0.1	0.0

4.3.2 Nitrided Ferritic Stainless Steel for 2 hours in Ammonia Environment at 600 ℃ (Slow cooling)

Figure 4 12: SEM-EDX of Nitrided FSS in Ammonia environment for 2 hours at 600℃ follows by slow cooling.

Table 4 7: Chemical Composition of Nitrided FSS in Ammonia environment for 2 hours at 600℃ follows by slow cooling.

Point		**80µ**	**160µ**	**240µ**	**320µ**	**400µ**
Chemical Composition (wt %)	Iron (Fe)	83.9	85.1	84.8	84.0	83.8
	Nitrogen (N)	**3.0**	**2.4**	**2.4**	**2.6**	**2.3**
	Chromium (Cr)	8.9	9.2	8.8	9.0	9.4
	Manganese (Mn)	1.0	0.7	0.9	1.0	0.8
	Molybdenum (Mo)	1.1	1.2	1.3	1.3	0.7
	Nickel (Ni)	1.0	0.5	0.8	1.4	1.7
	Silicon (Si)	0.8	0.5	0.8	0.5	0.8
	Phosphorus (P)	0.2	0.2	0.2	0.2	0.3
	Titanium (Ti)	0.0	0.0	0.0	0.0	0.0
	Sulphur (S)	0.1	0.2	0.0	0.0	0.2

4.3.3 Nitrided Ferritic Stainless Steel for 8 hours in Ammonia Environment at 600 ℃ (Slow cooling)

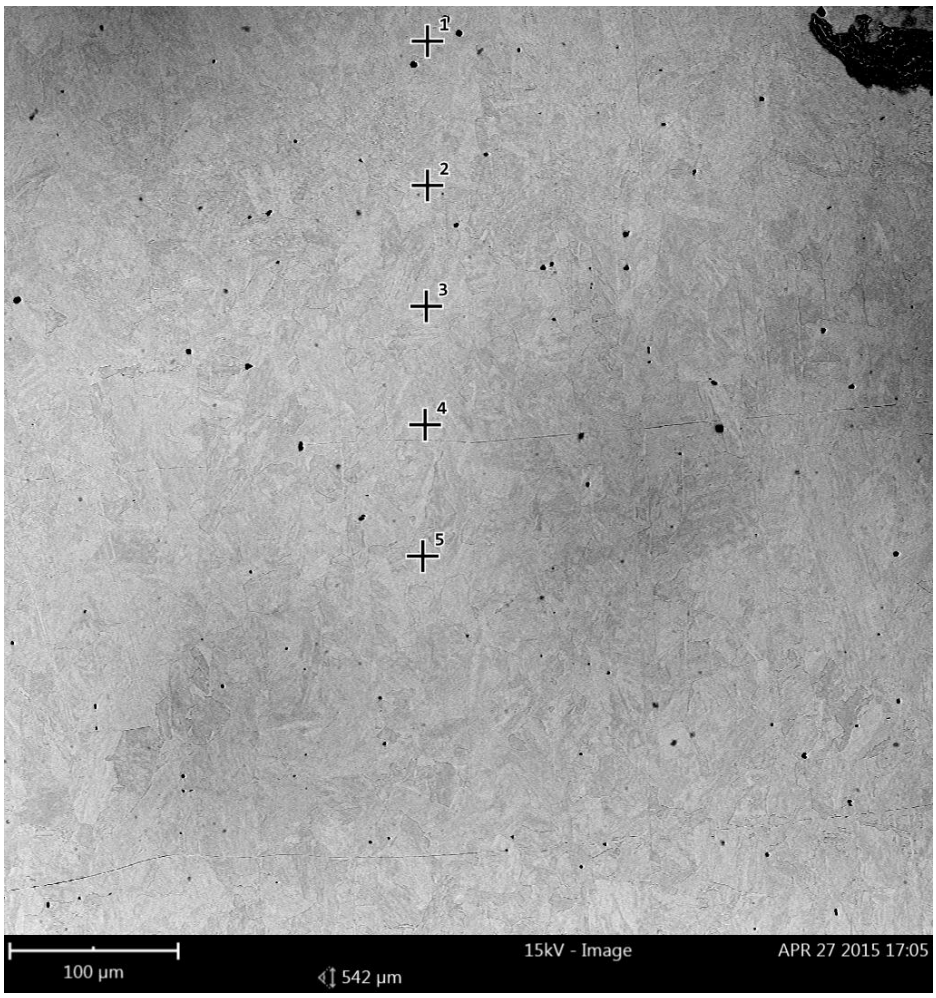


Figure 4 13: SEM-EDX of Nitrided FSS in Ammonia environment for 8 hours at 600℃ follows by slow cooling.

Table 4 8: Chemical composition of Nitrided FSS in Ammonia environment for 8 hours at 600℃ follows by slow cooling.

Point		**80µ**	**160µ**	**240µ**	**320µ**	**400µ**
Chemical Composition (wt %)	Iron (Fe)	84.7	83.0	84.3	84.4	84.0
	Nitrogen (N)	**2.9**	**3.2**	**3.0**	**2.8**	**3.0**
	Chromium (Cr)	8.8	9.0	8.7	8.9	8.5
	Manganese (Mn)	1.1	1.0	1.2	1.0	1.4
	Molybdenum (Mo)	0.2	1.4	0.3	0.5	0.7
	Nickel (Ni)	0.7	1.6	1.1	1.4	1.2
	Silicon (Si)	0.6	0.6	0.6	0.6	0.8
	Phosphorus (P)	0.3	0.2	0.3	0.2	0.2
	Titanium (Ti)	0.2	0.0	0.0	0.0	0.0
	Sulphur (S)	0.5	0.0	0.5	0.2	0.2

4.3.4 Nitrided Ferritic Stainless Steel for 24 hours in Ammonia Environment at 600 ℃ (Slow cooling)

Figure 4 14: SEM-EDX OF Nitrided FSS in Ammonia environment for 24 hours at 600℃ follows by slow cooling.

Table 4 9: Chemical composition of nitride FSS in Ammonia environment for 24 hours at 600℃ follows by slow cooling.

Point		**80µ**	**160µ**	**240µ**	**320µ**	**400µ**
Chemical Composition (wt %)	Iron (Fe)	82.9	84.3	83	83.5	83.8
	Nitrogen (N)	**3.2**	**3.0**	**3.2**	**2.7**	**3.0**
	Chromium (Cr)	8.8	8.7	8.7	8.8	8.3
	Manganese (Mn)	1.1	0.9	1.2	1.0	1.1
	Molybdenum (Mo)	1.7	0.4	1.6	1.7	0.8
	Nickel (Ni)	1.0	0.9	1.3	1.3	1.8
	Silicon (Si)	0.7	0.9	0.8	0.7	0.7
	Phosphorus (P)	0.4	0.4	0.2	0.3	0.3
	Titanium (Ti)	0.2	0.0	0.0	0.0	0.0
	Sulphur (S)	0.0	0.5	0.0	0.0	0.2

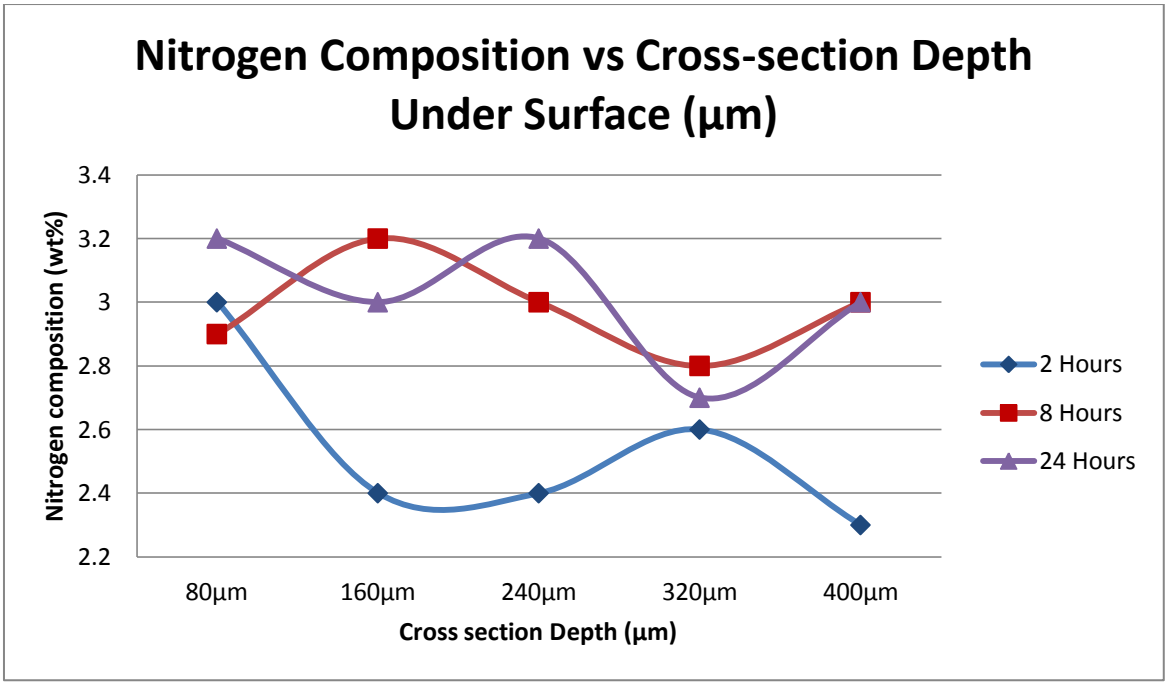


Figure 4 15: Cross Section Particle Concentration of nitride Ferritic Stainless Steel

Table 4 10: Average Nitrogen composition of nitride FSS in Ammonia environment for 2 hours, 8 hours and 24 hours

Nitriding Hours	Unitrided	2 Hours	8 Hours	24 Hours
80µm	0.0	3	2.9	3.2
160µm	0.0	2.4	3.2	3
240µm	0.0	2.4	3	3.2
320µm	0.0	2.6	2.8	2.7
400µm	0.0	2.3	3	3
Average Nitrogen Composition	0.0	2.540	2.980	3.020

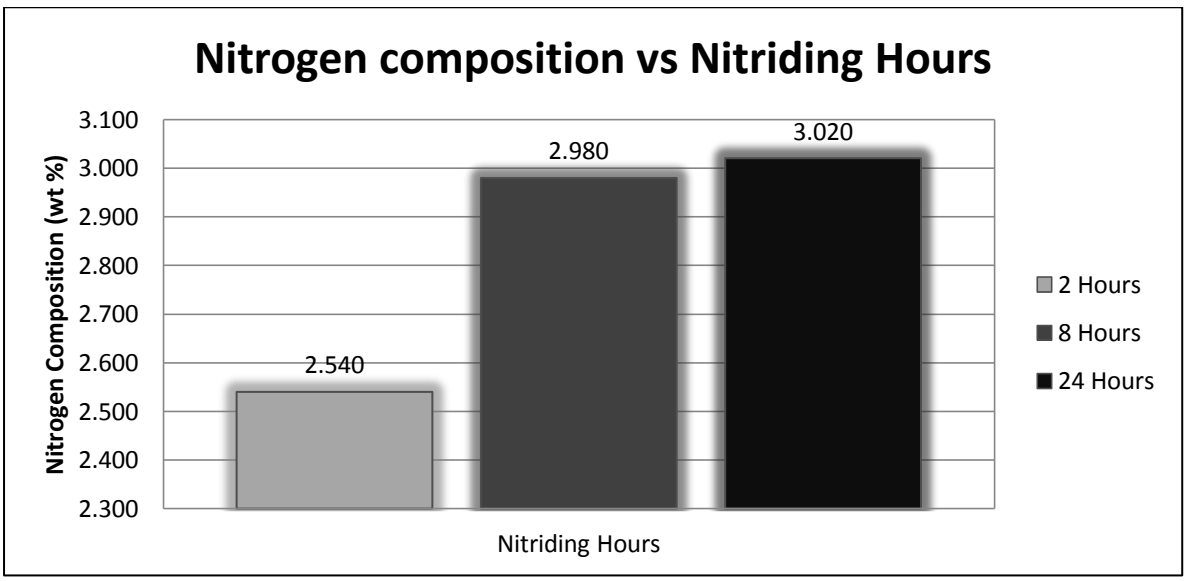


Figure 4 16: Nitrogen compositions at different nitriding time

Figure 4.11 shows SEM-EDX of Unitrided Ferritic stainless steel, the reading is pointed at random location to investigate particle concentration inside the steel. Meanwhile, Figure 4.12, Figure 4.13 and Figure 4.14 shows SEM-EDAX of nitride FSS at different period of time. The reading is taken every 80µm, to investigate the nitrogen particle diffusion rate inside the steel. From Figure 4.15, we can conclude that the nitrogen diffusion rate decreases with cross-section depth under the surface.

Figure 4.16 shows average particle concentration of nitrided Ferritic stainless steels at 2 hours, 8 hours and 24 hours. From the figure we can clearly see that overall nitrogen particle concentration increases with nitriding time. Increasing in composition of nitrogen will increase the dislocation barrier, fill up the space available, finally will increase the overall hardness of the material, as shown in Figure 4.17 below.

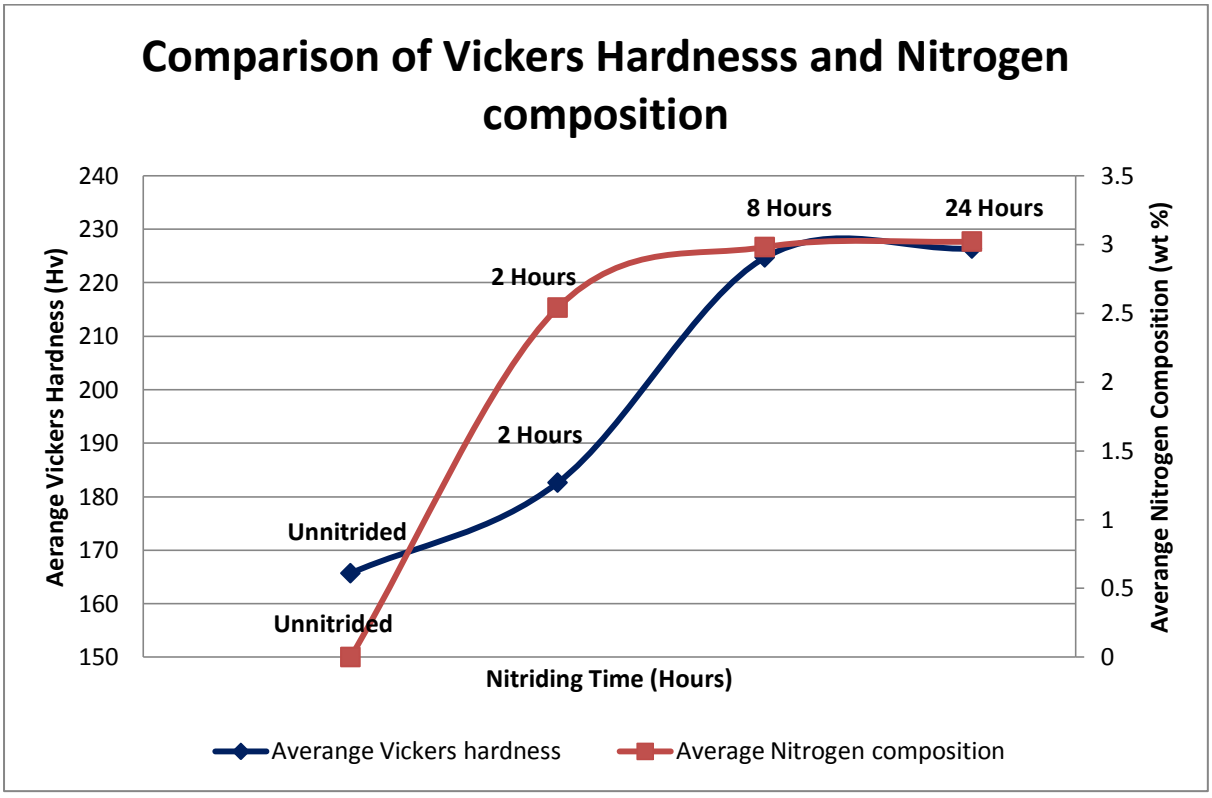


Figure 4 17: Comparison of Vickers hardness and Nitrogen composition with Nitriding time

CHAPTER 5
CONCLUSION AND RECOMMENDATIONS

5.1 Conclusions

Unitrided Ferritic Stainless steel grade 430L has bigger sizes of grain compare to Nitrided Ferritic Stainless Steel, this is due to heat treatment during manufacturing process. Heating at higher than crystalline temperature, small grain merges each other forming bigger size of grain. The number of grain boundaries increasing with time of nitriding respectively, thus increase the hardness value of Nitrided FSS.

The hardness value for untrided, 2 h nitride, 8 h and 24 h were 165.67 Hv, 182.6 Hv, 224.69 Hv and 226.34 Hv, respectively. Thus the highest hardness was determined for sample nitride at 24 h. The hardness increase by 30 % compares to untrided sample. The hardness increases at decreasing rate, due to high concentration of nitrogen inside the steel.

Micro hardness of materials decreases with depth under the surface, the weakest point is located at the centre of the material which is the deepest depth diffusion rate can diffuse. This due to the concentration of metal particle increases along the diffusion path, reducing the space for diffusion rate to occur.

The concentration of Nitrogen inside the metal reduces with the depth under the surface. Nitrided Ferritic stainless steels at 24 hours have the highest nitrogen content compare to Nitrided Ferritic stainless steel at 8 hours and 2 hours. Unnitrided Ferritic stainless steels have the lowest nitrogen content. The nitrogen composition of nitrided FSS increases at decreasing rate with respect to nitriding time. The micro hardness of FSS depends with microstructure of the material. The smaller the grain size, the harder the materials. The strength of material increases proportionally with increasing the composition of nitrogen in the steel. Therefore, the objectives of the research have been achieved.

5.2 Recommendation

Some improvements need to be done in order to obtain more detail and more precise result. Recommendation for the result improvement is effect of nitriding towards corrosion rate of the steel, and comparison effects of nitriding in argon environment, nitriding in nitrogen environment and also nitriding in high temperature gas nitriding.

CHAPTER 6
REFERENCES

[1] Kenneth G. Bundinski, Michael K. Bundinski, Engineering Materials Properties and Selection 9th Ed, New Jersey: Pearson , 2010.

[2] J.M. Aquino, C.A. Della Revere, S.E. Kuri, "Intergraular corrosion susceptibility in supermartensitic stainless steel weldments," in *Corrosion Science Volume 51*, Sao Carlos Fereral University, Material Engineering Department, Rodovia Washinton Luis km 235. Sao Carlos, Sp, Brazil, Elsevier, 2009, pp. 2316 - 2332.

[3] "Stainless steels family. International Stainless steels forum.," [Online]. Available: http://www.worldstainless.org/Files/issf/non-image-files/PDF/TheStainlessSteelFamily.pdf. [Accessed 30 October 2014].

[4] A. M. Ltd, "Stainless steel 1.4016 Sheet and Plate," Aalco The UK's Largest independest multi-metals stockholder, [Online]. Available: http://www.aalco.co.uk/datasheets/Stainless-Steel-14016-Sheet-and-Plate_95.ashx. [Accessed 1 April 2015].

[5] T. S. S. C. Ltd, "AISI 430 Ferritic Stainless Steel". Thailand.

[6] D. Pye, "An Introduction to Nitriding," *Practical Nitriding and Ferritic Nitrocarburizing (ASM International),* no. ISBN: 978-0-87170-791-8, pp. 1-12, Desember 01, 2003.

[7] Nor Nurulhuda Md. Ibrahim, Patthi Hussain, Mokhtar Awang, "Microstructure and Hardness of High Temperature Gas Nitrided AISI 420 Martensitic Stainless Steel," in *MATEC Web Conferences 13, 04017*, Universiti Teknologi Petronas, Bandar Seri Iskandar, 31750 Tronoh, Perak, Malaysia, 2014.

[8] W. K. W. Kamarudin, Study the effect of nitriding on the hardness properties of Duplex Stainless Steels, Tronoh, Perak, Malaysia: Universiti Teknologi PETRONAS, 2014, pp. 5-6.

[9] Yun Tao Xi, Dao Xin Liu, Dong Han, "Improvement of Corrosion and wear resistance of AISI martensitic stainless steel using plasma nitriding at low temperature," *Surface and Coting Technology,* no. 202, pp. 2577 - 2583, 2008.

[10] J.H. Sung, J.H. Kong, D.K. Yoo, H.Y. On, D.J. Lee, H.W. Lee, "Phase changes of the AISI 430 Ferritic Stainless Steels after High Temperature Gas Nitriding and Tempering Heat Treatment," *Materials Science and Engineering ,* vol. A , no. 489, pp. 38-43, 2008.

[11] A. N. Nazlan, Investigation of fetigue resistance of Nitrided Duplex Stainless Steel at low temperature, Universiti Teknologi Petronas, Bandar Seri Iskandar, Tronoh, Perak: Mechanical Engineering Department, Universiti Teknologi Petronas, 2015.

[12] W.D. Callister, D.G. Rethwisch, "Dislocations and Strengthing Mechanism," in *Materials Science and Engineering 8th Edition*, John Wiley & Sons (Asia) Pte Ltd, 2011, pp. 197-233.

[13] M. Roy, Vickers Hardness Measurements: Practice and Calibration Check, Vanconver, B.C. Canada: The University of British Columbia, July 22, 2010.

[14] T. Udomphol, "Hardness Test," Suranaree University of Technology , May - August 2007.

[15] Hassan R. S. Mahmoud, Syafiq A. Yusoff, Azman Zainudiin, Patthi Hussain, Mokhtar Ismail, Effective Duration of Gas Nitriding Process on AISI 316L for the formation of a desired thickness of surface nitrided layer, Universiti Teknologi PETRONAS, Malaysia: EDP Science, 2014.

[16] G. V. Voort, "VACAERO International Inc," 2015. [Online]. Available: http://vacaero.com/information-resources/metallography-with-george-vander-voort/1061-identification-of-phases-in-stainless-steels-by-etching.html. [Accessed 13 April 2015].

[17] P.-W. B.V, Phenom Pro-x User guideline, Dillenburgstraat 9E,5652 AM Eindhoven,The Netherlands: Phanom-World B.V. (www.phenom-world.com), 2014.

[18] A. M. A. Division, "EDAX, Smart Insight," 2015. [Online]. Available: http://www.edax.com/periodic-table/index.aspx#thumb. [Accessed 13 April 2015].

[19] T. Bergamo, "Effect of Nitriding on High Temperature Corrosion of Ferritic Stainless Steel," Department of Materials and Manufacturing Technology, Chalmers University of Technology Goteborg, Sweden, 2013.

[20] ioWiki, "ioWiki," 27 October 2013. [Online]. Available: http://iowiki.com/case-hardening-processes/. [Accessed 9 April 2015].

CHAPTER 7
APPENDIX

The formulae of Vickers Hardness as bellow:

$$\text{A} = \frac{d^2}{2\sin(\alpha/2)} = \frac{d^2}{1.8544}$$

Therefore, the Vickers Hardness, H_v in kg/mm^2 is equal to:

$$H_v = \frac{F}{A} = \frac{2p\sin(\alpha/2)}{d^2} = \frac{1.8544F}{d^2}$$

Where, P is the load in kgf. The base SI unit:

$$H_v = \frac{F}{A} = \frac{1.8544F}{d^2}$$

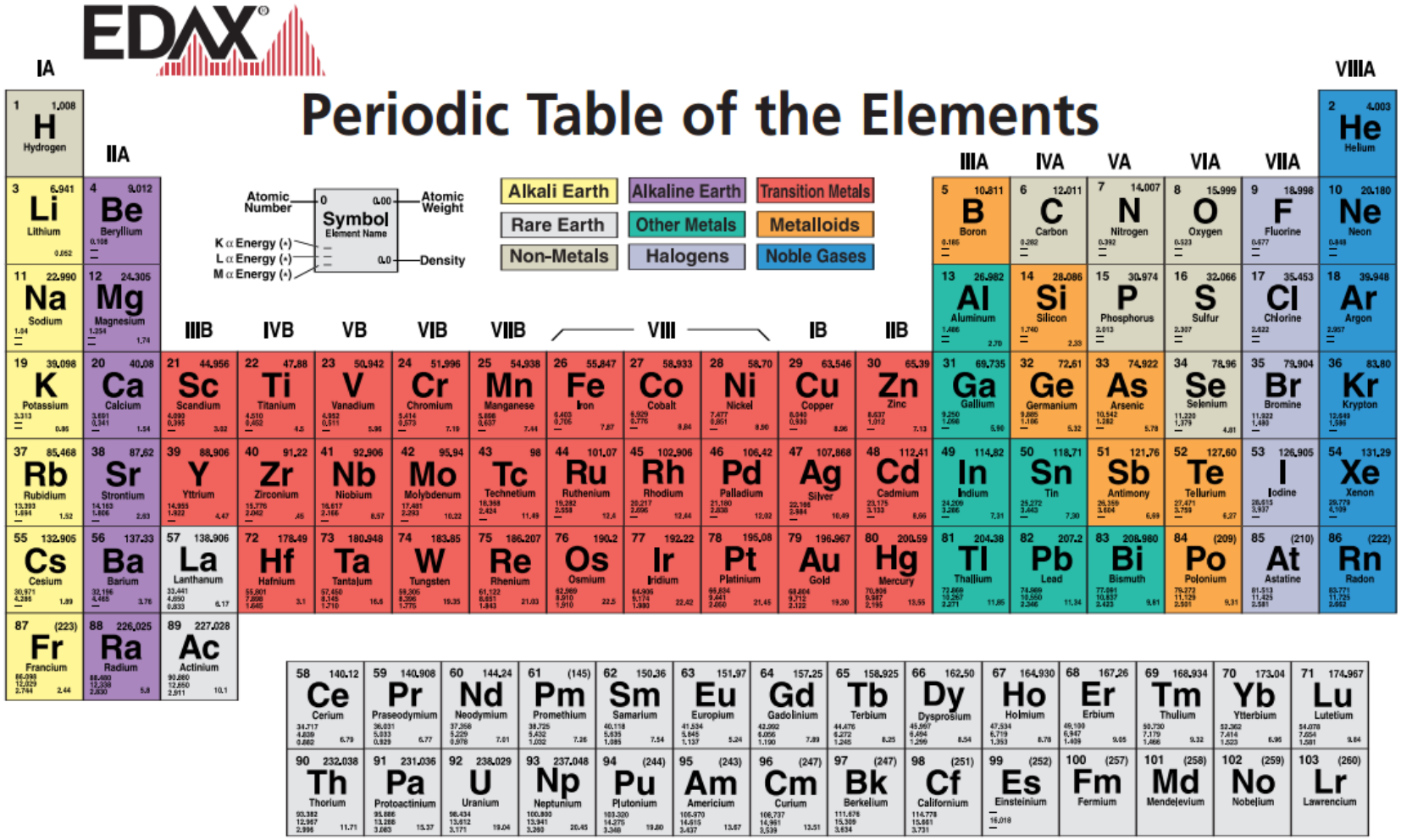
A as a function of depth h is given by:

$$\text{A} = 4h^2 tan^2(\alpha/2)$$

The hardness is a function of this depth h is:

$$H_v = \frac{F}{24.5h^2}$$

[13]



AMETEK

EDAX Inc.
Corporate Headquarters
91 McKee Drive • Mahwah, NJ 07430
Tel: 201-529-4880
Fax: 201-529-3156

EDAX BV
Regional Office
Tilburg, The Netherlands
Tel: +31-13-536-4000
Fax: +31-13-535-6279

EDAX Japan KK
Regional Office
Tokyo, Japan
Tel: +81-3-3740-5172
Fax: +81-3-3740-5307

EDAX Asia Pacific
Regional Office
Kowloon, Hong Kong
Tel: +852-2626-0971
Fax: +852-2699-4114

TSL

Web: www.edax.com • Email: info@edax.com

Figure 7 1: Periodic Table of element with energy dispersion of each element. [18]

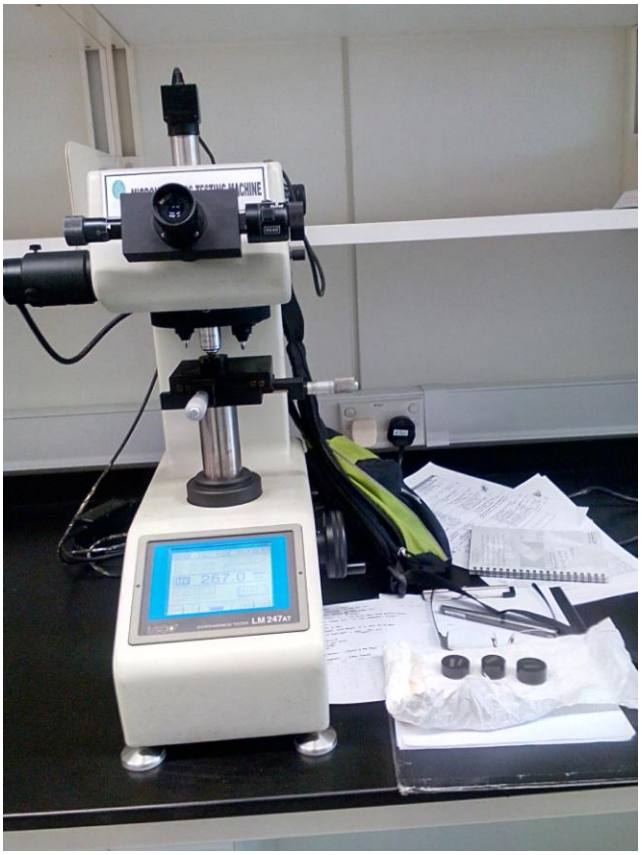


Figure 7 3: LECO Micro hardness Tester LM247 AT

Figure 7 2: LEICA Optical Microscope DMLM with DFC425 Image converter

Figure 7 4: Furnace equipped with gas nitriding accessory

SAMPLE PREPARATION STEP BY STEP

1. PLACE A CARBON ADHESIVE ON AN EMPTY ALUMINIUM PIN STUB

2. REMOVE TOP SIDE ADHESIVE

3. PLACE A SAMPLE

4. PLACE THE SAMPLE IN THE HOLDER

5. ADJUST THE HOLDER HEIGHT

6. PLACE THE HOLDER IN THE PHENOM

PHENOMWORLD

Figure 7 5: Sample Preparation of SEM-EDX [17]

PHENOM SAMPLE PREPARATION TIPS

THE FOLLOWING GUIDELINES SHOULD BE FOLLOWED CAREFULLY IN ORDER TO GET THE BEST IMAGES AND PREVENT DAMAGE TO THE PHENOM™ DESKTOP SCANNING ELECTRON MICROSCOPE. ADDITIONAL INFORMATION CAN BE FOUND IN THE PHENOM USER MANUAL.

1. NEVER PREPARE A SAMPLE IN THE PHENOM SAMPLE HOLDER

Loose particles/sample material can end up in the bottom of the holder, which can then contaminate the SEM column.

2. SAMPLES NEED TO BE DRY

Wet samples may outgas under vacuum which can cause imaging problems as well as contamination of the SEM column or a reduction of the source lifetime. Wet samples can only be imaged by freezing them using the optionally available Temperature Controlled Sample Holder.

3. SAMPLES MUST BE FIRMLY ATTACHED

Verify that your sample is firmly attached to the sample stub. Colloidal graphite, silver paint, or a conductive adhesive pad can be used to affix the sample to the sample stub.

4. POWDER SAMPLES

After firmly fixing powder samples to the stub, spray with clean dry compressed gas to remove any loose particles or debris.

5. CHARGING SAMPLES

In case of charging samples, the Charge Reduction Sample Holder can be used to minimize charging effects on non conductive samples. For samples that still display a charging effect under the SEM, a coating of gold or other heavy metal will improve the imaging capability.

Figure 7 6: Precaution during Sample Preparation [17]